



ADVANCES IN HETEROCYCLIC CHEMISTRY

Volume 55

Alan R. Katritzky

Advances in

Heterocyclic Chemistry

Volume 55

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Advances in

HETEROCYCLIC CHEMISTRY

Edited by

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Preface

Martin Bryce, Jan Becher, and Birgitte Fält-Hansen provide a survey of the behavior of thioaldehydes and their seleno and teleuro analogues, and of thionitroso compounds and their seleno analogues, as heterodie-nophiles. This is a subject which has exploded in recent years with three quarters of the references from the last decade.

L. I. Belen'kii and N. D. Kruchkovskaya have updated their survey of the literature of heterocyclic review articles. This is the fourth installment in this series: previous overviews of heterocyclic reviews appeared in Volumes 7 (literature up to 1965), 25 (literature 1966–1978) by S. M. Weeds and P. M. Jones with your editor, and Volume 44 (literature 1979–1987) by L. I. Belen'kii. A particular feature of this overview is its coverage of the Russian language literature as well as Western sources.

Heinrich Wamhoff and Jörg Dzenis discuss the synthesis, structure, and reactions of uracils with particular emphasis on their utility in heterocyclic synthesis.

Finally, S. Arai and M. Hida review polycyclic aromatic nitrogen cations containing bridgehead (ring fusion) nitrogen atoms. The chemistry of quinolizinium salts was covered by Thyagarajan in Volume 5 of our series back in 1965, and although other partial reviews are available, we now have for the first time a modern comprehensive treatment.

A. R. KATRITZKY

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Heterocyclic Synthesis Using New Heterodienophiles

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I. Overview

Heterodienophiles represent a very important class of compounds that are enjoying considerable use in organic chemistry, especially for the formation of heterocycles via cycloaddition reactions. A broad survey of the chemistry of a large number of heterodienophiles has been published (87MI1). This chapter focuses on the systems of the general formula $RX=Y$ (where $X = CH$ or N ; $Y = S, Se, \text{ or } Te$; $R = \text{any substituent}$). Most of these systems are transient intermediates that cannot be isolated. It is only in the last few years that the chemistry of these systems has been explored in a systematic way. Within this series of compounds, thioaldehydes, $RCH=S$, are by far the most widely used in synthesis. Preparative routes to selenoaldehydes and telluroaldehydes have invariably been developed in the light of previous work on the analogous thio systems. The heavier chalcogens form the least stable systems, $RCH=Y$, and good evidence for the generation of a telluraldehyde was not reported until 1989.

A wide range of thionitroso compounds, $RN=S$, are now firmly established as reactive intermediates, yet many features of their chemistry remain unexplored. Selenonitroso compounds, $RN=Se$, on the other hand, have been observed only by spectroscopic techniques at very low temperatures, and telluronitroso compounds remain undetected.

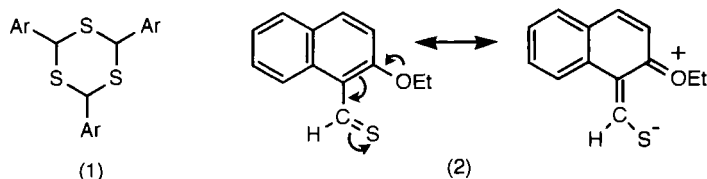
II. Thioaldehydes, $RCH=S$

A. GENERAL CONSIDERATIONS

The synthesis and properties of thioaldehydes have been reviewed by Russian workers (90UK649). Only recently in the 150-year history (46CRV1; 57MI1; 79MI1) of the chemistry of thioaldehydes has the research emphasis in this area been on anything but their generation. Most attempts to prepare thioaldehydes directly from the corresponding aldehydes using H_2S led to trimers or oligomers. Simple thioaldehydes were described as being so unstable that their monomeric forms could be detected only by spectroscopy or by trapping as Diels–Alder adducts. Later, the first stable thioaldehydes were isolated as monomers. However, in the early 1980s, several groups reported more general preparations of thioaldehydes and described the use of thioaldehydes in organic synthesis.

In 1841, Laurent obtained thiobenzaldehyde decamer from reacting oil of bitter almonds (which is mostly benzaldehyde) with ammonium sulfate (1841LA320), although the size of the oligomer was not established until

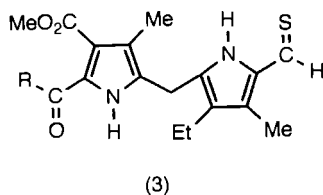
50 years later (1891CB1431). In 1876, Klinger first identified the trimeric product from the reaction of benzaldehyde with H_2S and HCl (1876CB1893). Later thiobenzaldehyde was described as 1,3,5-trithian derivative (**1**) in two geometric forms, an α -form and a β -form (47JCS693; 52JA2878). Even more modern sulfurating agents such as hexamethyldisilylthiane (79ZOB1084) and boron trisulfide (82JA3104) merely afforded trimeric products on reaction with aldehydes.



The unstable thioaldehyde group, like other reactive heterodienophiles, can be stabilized either thermodynamically or kinetically. Thermodynamic stabilization can result from conjugation, while kinetic stabilization is possible via the introduction of sterically bulky groups protecting the thioformyl group. Up to 1980, there have been numerous reports of stable, isolable thioformyl-containing compounds, which are stabilized via conjugation to an aromatic system or vinylogous conjugation to a heteroatom or both. Claims of thioaldehyde isolation have appeared for compounds that are really only vinylogous thioformamides. NMR analysis of these compounds showed a high degree of conjugation (72OMR421).

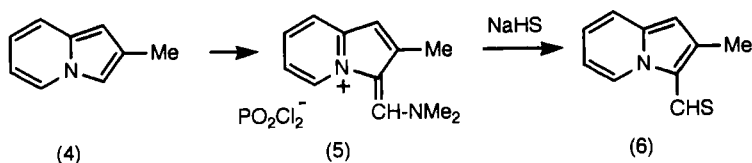
One of the first thioaldehydes stabilized by an aromatic system and by heteroatoms was 2-ethoxynaphthalene-1-thiocarboxaldehyde (**2**), which was isolated as a trimer by reacting the corresponding aldehyde with H_2S in alcohol under acidic conditions. By heating the trimer, it was possible to distill a small amount of monomer, which, however, polymerized after a few hours at room temperature (37JA1721).

In 1960, Woodward *et al.* obtained the first heterocyclic thioaldehyde (**3**) by reacting the hydrobromide of the corresponding *N*-(ethylformimino) compound with H_2S in the presence of base (60JA3800). This thioaldehyde (**3**) is stabilized by conjugation to N, and it was a famous precursor in the total synthesis of chlorophyll.

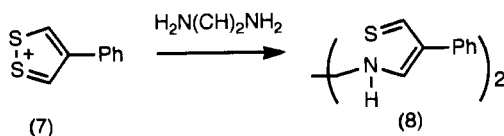


B. PREPARATIVE METHODS AND REACTIONS

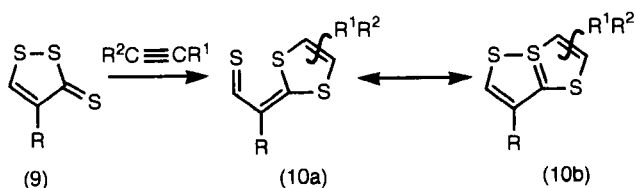
In the first general synthesis of heterocyclic thioaldehydes, McKenzie and Reid described a new application of the Vilsmeier–Haack reagent [66CC401; 70JCS(C)145]. After reacting indolizine (4) with POCl_3 and dimethylformamide (DMF), the Vilsmeier–Haack salt (5) was hydrolyzed to the thioaldehyde (6) in aqueous NaHS . This method successfully gave stable indolizines, which were later followed by 4-thioformylindenothiazoles [69JCS(C)913] and pyrrolo-isothiazoles [73JCS(P)1657].



1,2-Dithiolium cation (7) has been used for synthesizing enaminothioaldehydes (8), which were precursors to macrocycles (73JA613). The thioformyl group of 8 is stabilized through vinylogous conjugation with a nitrogen lone pair.



McKinnon and Buchschriber used 1,2-dithiol-3-thiones (9) and substituted acetylenedicarboxylates in the synthesis of thioaldehydes in which the thioformyl group is in vinylogous conjugation with a sulfur heterocycle (71CJC3299). These thioaldehydes (10) can be described as the structure 10b, but X-ray results show that structure 10a is a more correct representation [74ACS(B)964; 75BSF1435].



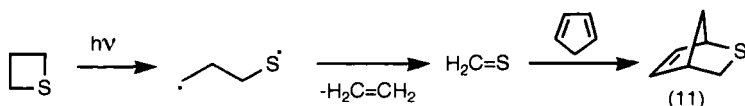
In the early 1980s, research into thioaldehydes was intensified after successful trapping by Vedejs *et al.* (80JOC2601; 82JA1445) and Baldwin and Lopez (82CC1029) of unstable thioaldehydes as their Diels–Alder adducts. It then became possible to work with this interesting functional group, whereas previously, extensive polymerization had thwarted attempts of synthesis. After this breakthrough, several groups have extensively studied the synthesis and chemistry of unstable thioaldehydes. Seven general methods reported since 1980 have been used in the synthesis of thioaldehydes (88Y GK1149):

- (1) Photolytic reactions of α -ketosulfides
- (2) Cleavage of thiosulfonates
- (3) Fragmentation of sulfur ylides
- (4) 1,2-Elimination reactions
- (5) Retro Diels–Alder reactions of cycloadducts
- (6) Reaction of metal–organic compounds with chalcogenoformates
- (7) Thermolysis of polymeric thioaldehydes ($\text{RCH}=\text{S}$).

Thioaldehydes used as reactive intermediates are mostly synthesized by methods (1)–(5), while stable thioaldehyde has been synthesized via method (6) or (7).

1. Photolytic Reactions of α -Ketosulfides

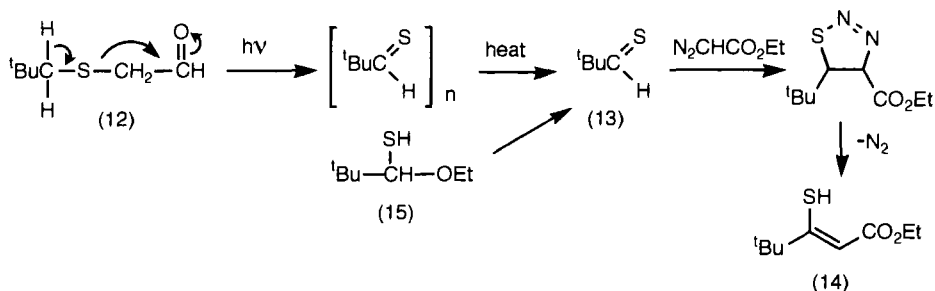
Several accounts of transient thioaldehydes generated from high-energy processes and detected spectroscopically or trapped as Diels–Alder adducts have appeared. Dice and Steer obtained thioformaldehyde from retro [2 + 2]-fragmentation by photolysis, and subsequent trapping afforded (**11**) (74CJC3518). This is the only report, before the work of Vedejs, that describes intermolecular Diels–Alder trapping of thioaldehydes. However the method is not preparatively useful.



Thioaldehydes with almost any substituents in the α -position can be generated according to Vedejs by photofragmentation of phenacyl sulfides (86JOC1556). This is a simple, effective, and mild reaction which can be performed in neutral media. Phenacyl sulfides are known to be unstable to visible and UV light (68CC700). Hogeveen and Smit had previously

reported the generation of polythioacetaldehyde and acetophenone from ethyl phenacyl sulfide (66RTC489).

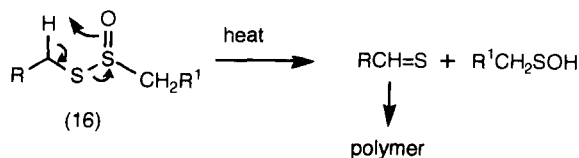
In 1983, the generation of 2,2-dimethylpropanethioaldehyde (thiopivaldehyde) (**13**), which was the first simple thioaldehyde to be stable in solution, was described (83JA1683). Thus, a solution at 0°C of sulfide (**12**) containing a diene is photolyzed by exposure to light, whereupon the generated thioaldehyde is trapped *in situ* to yield (**14**). The reaction mechanism is a Norrish type II, which involves a 1,5-shift to carbonyl oxygen and fragmentation of the C—S bond. The thioaldehyde itself is an insoluble white polymeric product. This polymer is depolymerized by heating to 250°C, and thiopivaldehyde, which is isolated as the liquid monomer by distillation, is stable for 16 hrs as a CHCl_3 solution at room temperature. Reactions of the monomer (**13**) with dienes and 1,3-dipoles proceed readily. For example, ethyl diazoacetate gave product **14**, presumably via an initial cycloadduct which loses nitrogen. Vedejs *et al.* have also generated thiopivaldehyde (**13**) by a different method (86JA2985). Butyl lithium and ethyl thioformate reacted to give hemithiolacetal (**15**), which fragmented to the thioaldehyde upon heating.



Numerous other reports also describe thermal (76JA6405; 82JA312) and photolytic (81TL4421) processes that give thioaldehydes as intermediates.

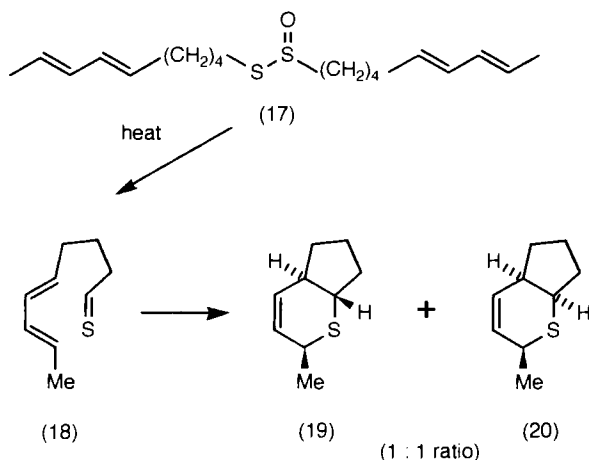
2. Cleavage of Thiosulfates

Another useful general route to thioaldehydes involves fragmentation of the S—S bond of thiosulfates. Block *et al.* showed that thiosulfates



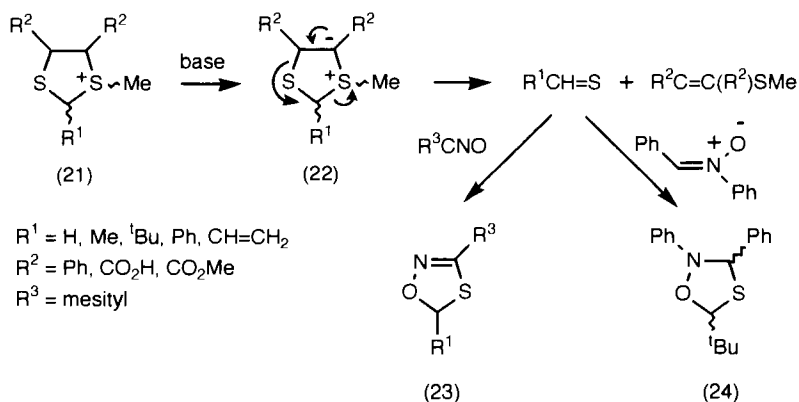
(16) rapidly fragment by heating, giving thioaldehydes (84JA8295) that sometimes polymerize under these conditions (72JA642, 72JA644; 86JA7045). A seleno variation has also been reported (87JA5549).

Baldwin and Lopez used fragmentation of thiosulfinate (17) to furnish transient thioaldehyde (18), which was trapped by intramolecular Diels–Alder reaction to yield bicyclic compounds (19) and (20) in equal amounts (82CC1029; 83T1487).



3. Fragmentation of Sulfur Ylides

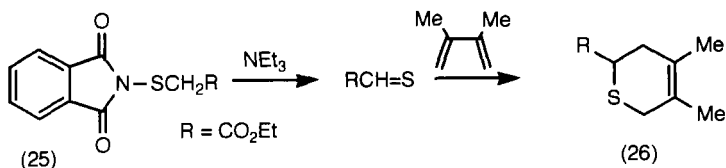
A route has been reported starting from 1,3-dithiolanes (21) that regio-specifically deprotonate in the presence of base to generate ylide (22), which spontaneously fragments to the thioaldehyde. Trapping reactions



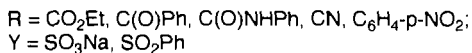
with 1,3-dipoles afford heterocycles **23** and **24**. The thioaldehyde can be regenerated from system **24** by being heated in toluene and trapped with dimethylbutadiene (85TL5265; 87MI2). (*cf.* Sec. II,B,5 for synthetically useful retro Diels–Alder reactions).

4. 1,2-Elimination Reactions

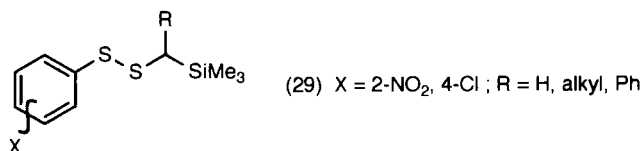
A general route to thioaldehydes is the base-induced 1,2-elimination of sulfonate derivatives, described initially by Kirby *et al.* In this reaction, phthalimide derivatives, e.g. (**25**), react with Et_3N to generate a thioaldehyde, which is subsequently trapped by dimethylbutadiene to yield product **26** [83CC423; 85JCS(P1)1541]. Other dienes used in this study include thebaine, cyclohexadiene and anthracene.



Thioaldehydes bearing an electron-attracting group, R, can be generated *in situ* starting from Bunte-salts (thiosulfate-S-esters) (**27**) and trapped by cyclopentadiene to yield isomeric adducts **28** with the endo isomer predominating (84CC922).

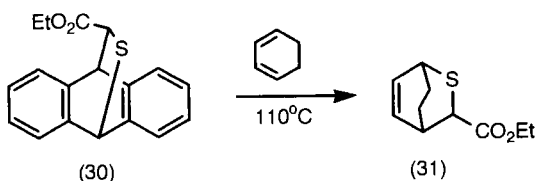


An analogous desilylation has been reported by Krafft and Meinke (85TL1947). Generation of the thioaldehyde occurs by fluoride-induced β -elimination of stabilized arylthiolate anions of α -silyl disulfides (**29**). The reaction is effective due to the stability of the arylthiolate leaving group. Again the evidence for thioaldehyde intermediacy is provided by Diels–Alder trapping with cyclopentadiene. Gas phase dehydrocyanation of thiocyanohydrins yields thioformaldehyde and thioacetaldehyde (91T4927).

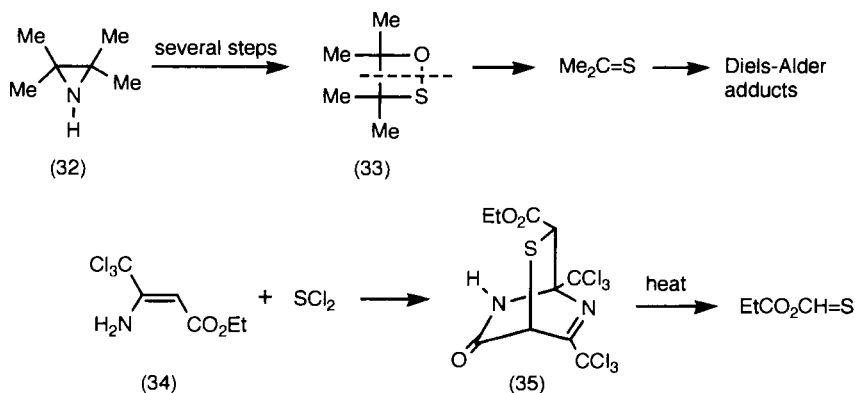


5. Retro Diels–Alder Reactions

Kirby and co-workers obtained alkyl thioacetate, which was trapped by a diene to give a Diels–Alder adduct, e.g. **30**. When this adduct was heated at 110°C in the presence of a new diene, a retro Diels–Alder reaction took place. The thioaldehyde thus generated gave a new adduct, **31**. Adduct **30** is, therefore, a thioaldehyde transfer reagent [85JCS(P1)1541].

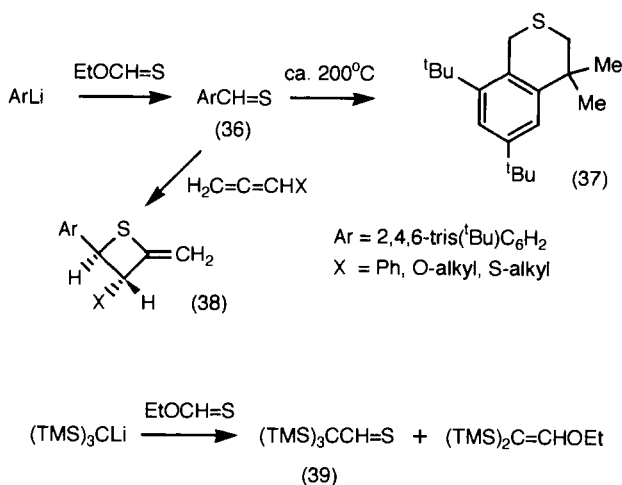


Lown *et al.* reported a simple retro [2 + 2] fragmentation of oxathietane (**33**) [prepared from tetramethylaziridine (**32**)] in aqueous medium, followed by trapping of thioacetone with anthracene (86JA3811, 86JOC2116). Lee *et al.* trapped ethyl thioacetate from retro Diels–Alder fragmentation of a bicyclic precursor (**35**), which was prepared in a fascinating reaction from the aminocrotonate (**34**) and sulfur dichloride (85JOC3216).

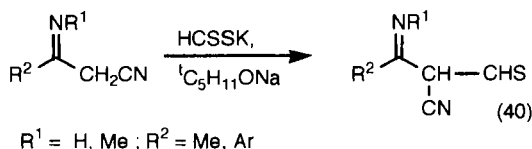


6. Metal–Organic Compounds with Thioformates

It is by this method, and method (7), that stable thioaldehydes have recently been isolated. Okazaki *et al.* isolated the first stable aromatic thioaldehyde, viz 2,4,6-tri-*tert*-butylthiobenzaldehyde (**36**) in 1982 (82CC1187; 83MI1; 84TL849; 87MI3). In 1987, tris(trimethylsilyl)ethanethioaldehyde (**39**) was obtained as a stable purple crystalline compound (87JA279). The thioformyl group in these two new thioaldehydes is kinetically stabilized by steric protection from the bulky *t*-butyl or trimethylsilyl (TMS) groups. The best route to **36** is the one-pot procedure from the arylbromide (56% yield). Compound **39** is obtained (16% yield) from tris(trimethylsilyl)methyl lithium and *O*-ethyl thioformate. Compound **36** is converted by heating or by photochemical reaction into benzothiolane (**37**) (84TL849). Thioaldehyde (**36**) undergoes photocycloaddition with substituted allenes to form thietanes (**38**) in high yield (84TL873).



Muraoka treated potassium dithioformate with β -iminonitriles in aprotic solvent [tetrahydrofuran (THF)] and sodium-1,1-dimethylpropanolate as base. The resulting α -cyano- β -iminothioaldehydes (**40**) are yellow or red stable monomers with well-defined melting points (82CL101). Thioformy-



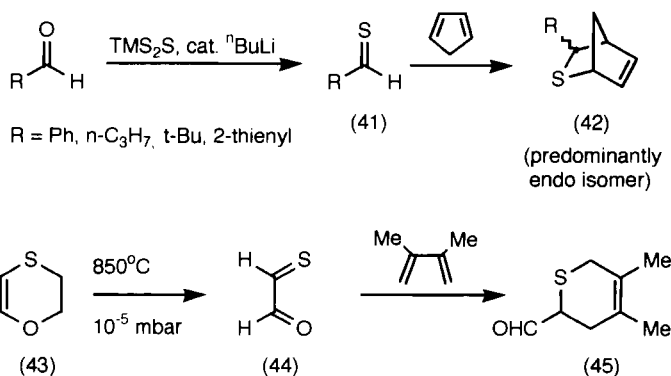
lation of enamines, under Vilsmeier–Haack conditions similar to those reported previously by Reid [69JCS(C)913] yielded stable enamino thioaldehydes (85CC1299).

7. Thermolysis of Polymeric Thioaldehydes

This method has been used by Vedejs *et al.* in the synthesis of thiopivaldehyde (**13**), as mentioned previously; the polymer is heated to 250°C, followed by depolymerization and isolation of the expected thiopivaldehyde as the monomer (86JA2985).

8. Other Methods

Segi *et al.* reacted some aldehydes with bis(trimethylsilyl) sulfide in THF with *n*-BuLi as catalyst. The thioaldehydes (**41**) thus formed were trapped with cyclopentadiene to afford adducts **42** as a mixture of endo and exo isomers (88JA1976). French workers have recently reported a gas-phase synthesis of thioxoethanal (**44**) from precursor (**43**): **44** was detected as an intermediate by photoelectron spectroscopy and by the formation of adduct **45** (90JOC2596).

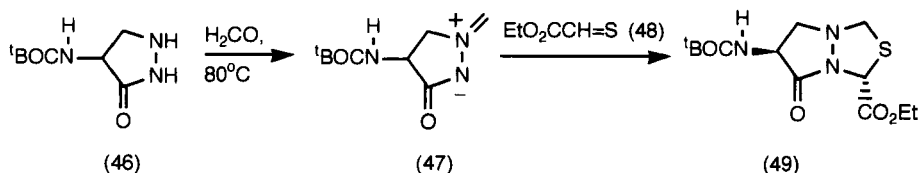


C. SYNTHETIC APPLICATIONS

1. Diels–Alder Reactions

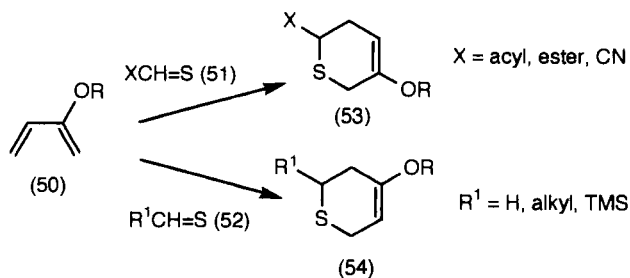
Thioaldehydes have been used as heterodienophiles in cycloadditions (Diels–Alder or 1,3-dipolar) mainly in connection with attempts to show the formation of the transient thioaldehydes by trapping *in situ*. During

the last few years, Vedejs *et al.* have extended the use of thioaldehydes as dienophiles in Diels–Alder reactions (83JA6999; 84JA573, 84JA4617; 87MI4; 88JA5452, 88JO2220, 88JO2226). In particular, intramolecular reactions occur readily (84JA4617). The total syntheses of zygosporin E and cytochalasin D (87MI4) and studies in the penicillin field (74CC47) have utilized thioformyl intermediates. Bicyclic system **49**, required as a part-structure of a pyrazolidinone antibacterial analogue, was prepared by reaction of ylide **47** (generated from compound **46**) with the thioaldehyde **48** liberated by retro Diels–Alder reaction of its anthracene adduct (88TL5061).



The high reactivity and polarizability of the thioformyl group makes this group a better dienophile than the corresponding aldehyde derivative. The electron-deficient thioaldehydes are the most reactive dienophiles: electron-donor substituents weaken the C=S double bond. Regiochemistry in the Diels–Alder adduct can be selected, depending on whether there is an electron-acceptor or electron-donor group in the α -position of the thioformyl group. Thioaldehydes (**51**) substituted with a π -electron acceptor group X attached to the thioformyl group undergo a relatively fast Diels–Alder reaction (83JA6999). With electron-rich dienes, such as 2-alkoxybutadiene (**50**), the reaction gives an adduct **53** with the typical Diels–Alder regiochemistry, i.e., with X para to the diene substituent.

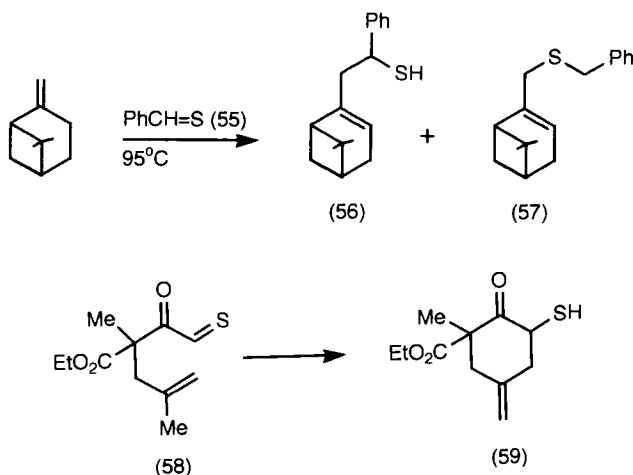
On the other hand, the regiochemistry is reversed for thioaldehydes (**52**) with an H atom or a donor group, R', in the α -position. The Diels–Alder



addition is now generally slower, and R^1 in the cycloadduct **54** is situated meta to the substituent on the diene. Diels–Alder reaction of thioaldehydes takes place stereochemically with preference for endo-addition over exo-addition. In this way, the thioformyl group can be used to prepare six-membered rings with control of regio- and stereochemistry.

2. -ene Reactions

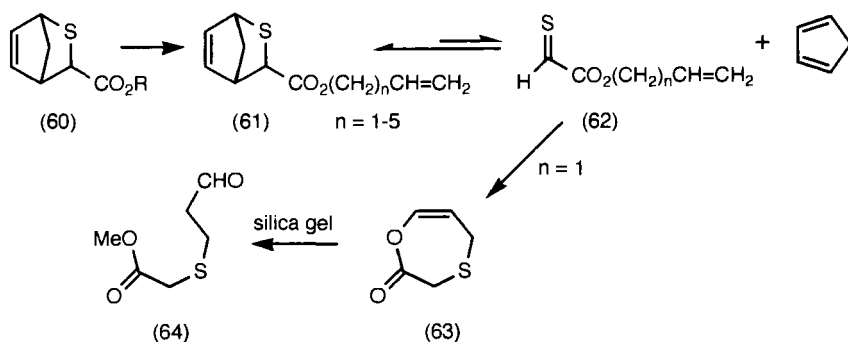
Thiobenzaldehyde (**55**), obtained by thermolysis of thiosulfinate, $\text{PhCH}_2\text{S(O)SCH}_2\text{Ph}$, gave by reaction with β -pinene, a 2 : 1 mixture of the two adducts **56** and **57**, which originate from different orientations of β -pinene in the -ene addition (83T1487). Intramolecular -ene reactions provide a range of interesting products, e.g., the carbocycle (**59**), which is derived from transient thioaldehyde (**58**) (84JA4617).



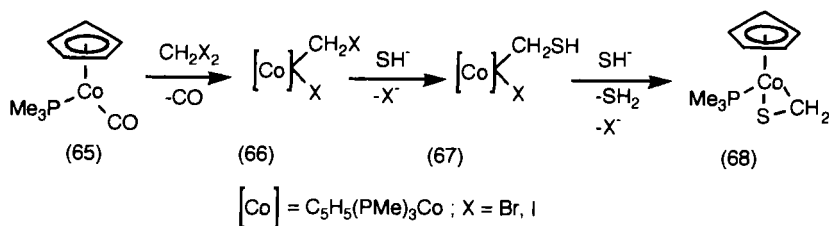
ω -Vinylalkyl esters of thioxoacetic acid, generated by retro Diels–Alder fragmentation of their cyclopentadiene cycloadducts, undergo intramolecular -ene reactions with the formation of new C—S bonds. The products are lactones, as exemplified in the sequence **60**–**63**. Ring opening of lactone **63** readily yields aldehyde **64** (88CC177; 90CC138).

3. Metal Complexes

Thioaldehydes have been used as ligands in metal complexes with osmium (76CC1044; 77CC901; 83JA5939) and rhenium (83JA1056). The first metal complexes with thio- (and seleno)-acetaldehyde as ligands were



obtained from Co and thioacetaldehyde. (85CB4229). The complexes **(66)** and **(67)** were obtained by reaction of NaSH (or NaSeH) and CH_2X_2 ($\text{X} = \text{Br}, \text{I}$) with $\text{C}_5\text{H}_5\text{Co}(\text{CO})\text{PMe}_3$.



III. Selenoaldehydes, $\text{RCH}=\text{Se}$

A. GENERAL CONSIDERATIONS

Hydrogen selenide is a stronger acid and a more reducing compound than hydrogen sulfide. It should, therefore, in principle be easier to generate selenoaldehydes from aldehydes and H_2Se than thioaldehydes from aldehydes and H_2S . However, selenoaldehydes are extremely difficult to isolate due to their high reactivity. Until recently, both selenoketones and selenoaldehydes had been elusive molecules. The initial synthesis by Barton's group in the mid 1970s of sterically crowded selenoketones [75CC539; 76JCS(P1)2079] was followed a few years later by chemical and spectroscopic studies on these systems [81TL4563; 85JCS(P1)107; 87TL3887] Reid *et al.* prepared and isolated stable systems akin to thioaldehyde **(6)** with the selenoformyl group attached to strongly electron-

donating heterocycles: these molecules are, in effect vinylogous seleno-amides with added stability from resonance delocalization of nitrogen lone pairs onto selenium [79JCS(P1)2334]. The synthetic procedure was based on previous work on analogous thioaldehydes. While several reports had described the pyrolytic formation and spectroscopic detection of seleno-formaldehyde and selenoacetaldehyde in the gas phase, (71JSP136; 77JCP1576; 84JA5406), no synthetically useful route to selenoaldehydes was available until 1986 (86JA1314).

The reactions of selenoaldehydes are limited almost exclusively to cycloadditions.

B. PREPARATIVE METHODS AND REACTIONS

There are four general methods of preparing selenoaldehydes:

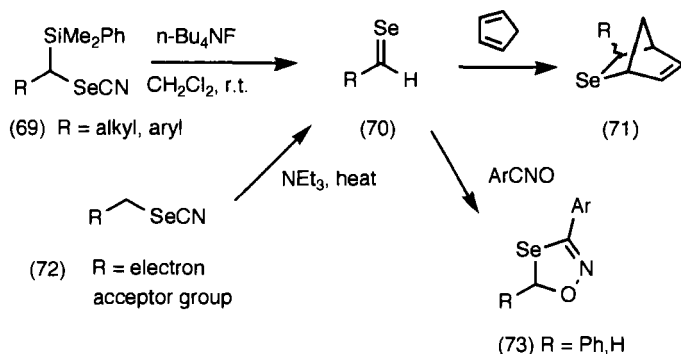
- (1) Fragmentation of α -silylselenocyanates
- (2) Fragmentation of selenenyl derivatives
- (3) Selenation of aldehydes
- (4) Reaction of sulfur or phosphorus ylides

1. Fragmentation of α -Silylselenocyanates

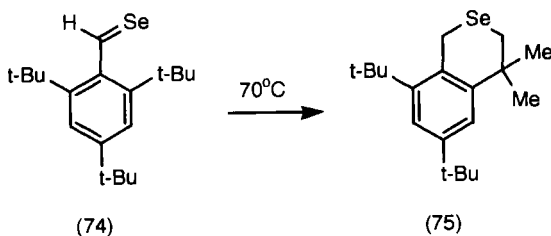
This method, developed by Krafft and Meinke, provided the first route to $\text{RCH}=\text{Se}$ systems that enabled the chemistry of this heterodienophile to be explored (86JA1314; 87TL5121; 88JA8671). Aryl- and alkyl-substituted derivatives **69** fragment on treatment with fluoride ions at room temperature to afford selenoaldehydes (**70**), which are trapped by dienes to give Diels–Alder adducts, e.g., **71** (predominantly endo isomer). Other dienes used include isoprene, 2-ethoxybutadiene, and 1,3-diphenylbenzo[c]furan (88JA8671).

These studies established that, in general, selenoaldehydes were more reactive as dienophiles than the corresponding thioaldehydes: selenals bearing electron-acceptor substituents (prepared from **72**, Section III,B,2) reacted faster than alkyl-substituted analogues. In reactions with unsymmetrical dienes, the cycloadditions of **70** exhibited typical ortho–para regiochemistry for electron-deficient selenals and meta regiochemistry for selenobenzaldehyde and selenoformaldehyde. Heterocyclic system **73** was obtained by reaction with mesitonitrile oxide.

This general method has recently been adopted by Okazaki for the preparation of 2,4,6-tri-*t*-butylselenobenzaldehyde (**74**), which is the first selenoaldehyde that can be isolated because of kinetic stabilization from

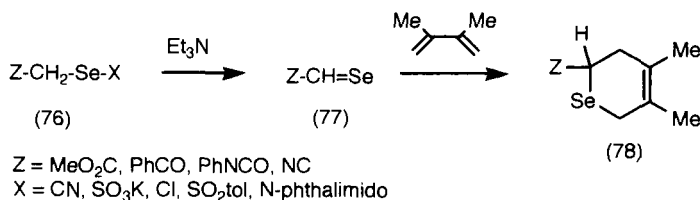


the bulky aryl substituent (89JA5949). Compound **74** is, however, far less stable than the analogous thioaldehyde (**36**); isomerization to yield **75** occurs at a lower temperature than that required to form **37**.



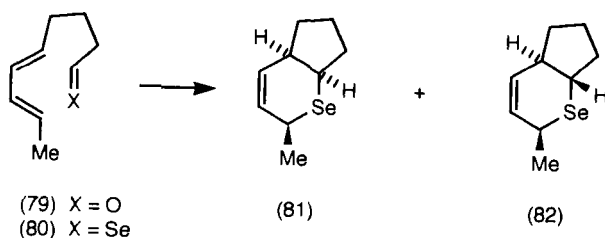
2. Fragmentation of Selenenyl Derivatives, ZCH_2SeX

This method, briefly mentioned previously (compound **72**) has been used primarily by Kirby and Trethewey to generate and trap highly reactive, electron-deficient selenoaldehydes [88JCS(P1)1913]. Precursors **76** were developed in the light of previous work on thioaldehydes. Base-induced 1,2-elimination from a range of systems of general formula **76** yields transient selenoaldehydes (**77**), which afford Diels-Alder adducts (e.g., **78**) with 2,3-dimethylbutadiene, thebaine, cyclopentadiene, anthracene, and 9,10-dimethylantracene. The adducts formed by the last three dienes dissociate at $80\text{--}120^\circ\text{C}$ with efficient regeneration of the selenoaldehyde by direct analogy with thioaldehydes (Section II,B,5). It seems that Diels-Alder reactions of selenoaldehydes are less stereoselective than those of thioaldehydes generated by the same route, which is consistent with the greater reactivity of the former species [88JCS(P1)1913].



3. Selenation of Aldehydes

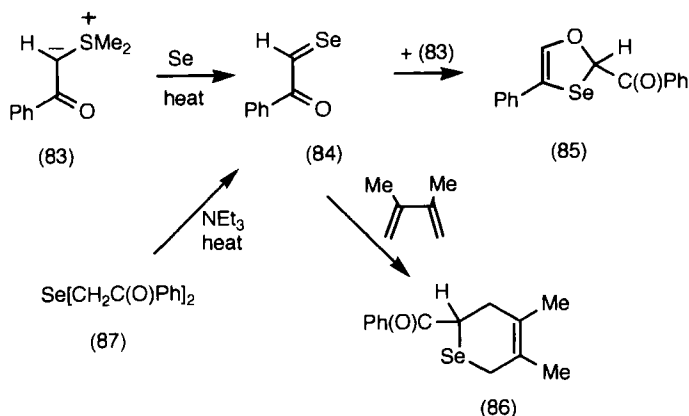
Early attempts to prepare selenoaldehydes by treatment of aldehydes with H_2Se under acidic conditions yielded oligomeric or polymeric selenides, possibly via self-condensation of selenoaldehydes [15JPR116; 67JCS(B)117]. In 1988, Japanese workers reported that the reaction of aldehydes with bis(trimethylsilyl)selenide in the presence of a catalytic quantity of butyl lithium afforded selenoaldehydes (88JA1976). This novel method has been exploited for the first intramolecular Diels–Alder reactions of selenoaldehydes (88TL6965). Dienal (**79**) was converted into dienselenal (**80**) and then into a mixture of stereoisomeric adducts **81** and **82**. The major product was *cis*-fused isomer **81**, derived from the endo transition state. These results compare favorably with intramolecular thioaldehyde reactions (Section II,B).



4. Reactions of Sulfur or Phosphorus Ylides

Nakayama and co-workers established that carbonyl-stabilized sulfur ylide (**83**) reacted with elemental selenium to afford 1,3-oxaselenole (**85**) in good yield (85TL2201). The reaction was presumed to proceed through the intermediacy of selenal (**84**). Subsequently, support for this mechanism has come from the isolation of adduct **86** when the reaction is performed in the presence of dimethylbutadiene (87TL4423). Other Diels–Alder reactions of intermediate α -oxoseleno-aldehydes and -ketones were also found

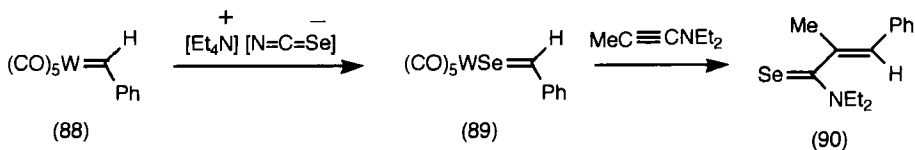
to proceed efficiently. Treatment of diketoselenide (**87**) with base provides an alternative method of generating **84** (88MI1).



Erker *et al.* reported that aryl- and alkyl-selenals are produced by reaction of phosphorus ylides with elemental selenium at 90°C and trapped with dimethylbutadiene and anthracene (88JA624).

C. ORGANOMETALLIC SELENOALDEHYDE COMPLEXES: SYNTHESIS AND REACTIVITY

Organometallic complexes of selenoaldehydes have been studied more widely than their thioaldehyde counterparts, largely in attempts to stabilize and confirm the existence of the $\text{RCH}=\text{Se}$ functional group. Several selenoaldehydes have been stabilized by coordination to tungsten and chromium complexes [84AG(E)726; 86AG(E)78; 87CC559]. For example, complex **88** yields complex **89**, the X-ray crystal structure of which reveals η^2 -coordination of the selenobenzaldehyde ligand. The $\text{C}=\text{Se}$ bond in complex **89** reacts with dimethylbutadiene and cyclopentadiene to yield metal-coordinated selenacycles. Alternatively, selenoamide (**90**) can be produced by reaction with an alkyne and decomplexation (87CC559).

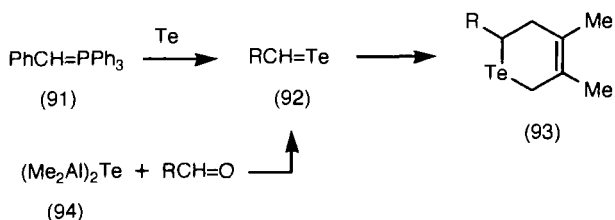


Selenoformaldehyde has also been described as a bridging ligand in binuclear osmium (83JA5939, 83JOMC53) and manganese complexes [83AG(E)314] and as a simple ligand in mononuclear rhodium complexes [83AG(E)316; 84AG(E)58].

IV. Telluraldehydes, $\text{RCH}=\text{Te}$

The lability of the carbon–tellurium double bond has frequently thwarted attempts to study both telluraldehydes and telluroketones. Tellurocarbonyl compounds stabilized by coordination to transition metals have been known since 1980 [80CC635; 83AG(E)314; 88JOM161]. However, free telluraldehydes were unknown until 1989 when two different synthetic routes were reported. Erker and Hock trapped tellurobenzaldehyde (**92**, $\text{R} = \text{Ph}$) generated by reaction of ylide (**91**) with tellurium powder; adduct **93** was obtained in low yield [89AG(E)179].

Segi and co-workers reported a more efficient procedure that furnished both phenyl- and alkyl-telluraldehydes (**92**). The new tellurating reagent (**94**) reacts directly with aldehydes in refluxing dioxane, and in the presence of a diene, the expected adducts **93** ($\text{R} = \text{Ph}$, $n\text{-Pr}$, and $t\text{-Bu}$) are formed in 44–62% yield (89JA8749). Telluroketones were prepared similarly and trapped with cyclopentadiene.



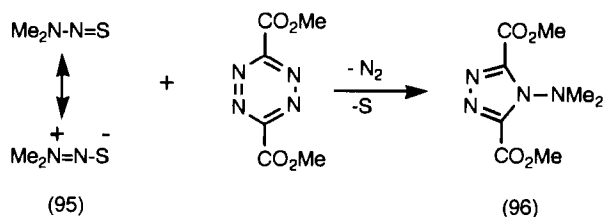
V. Thionitroso Compounds, $\text{RN}=\text{S}$

A. GENERAL CONSIDERATIONS

While the rich chemistry of nitroso compounds and sulfinylamines, $\text{RN}=\text{S}=\text{O}$, is routinely used in synthesis (87M11), the closely related thionitroso group, $\text{RN}=\text{S}$, has remained in obscurity. Although scattered literature reports of the intermediacy of thionitroso compounds have appeared from time to time since 1966 (66JA3842); efficient synthetic routes were not available until very recently. A striking difference between

the nitroso and thionitroso groups is the direction of polarization. Due to the positively charged sulfur, the $\text{RN}=\text{S}$ group tends to desulfurize forming sulfurdiimides, $\text{RN}=\text{S}=\text{NR}$. Like the foregoing $\text{RCH}=\text{Y}$ systems ($\text{Y} = \text{S}, \text{Se}, \text{and Te}$), thionitroso compounds should be thought of as highly reactive, transient species that cannot be isolated except under special circumstances.

Middleton established that reaction of 1,2-dialkylhydrazines with sulfur and reduction of sulfinylhydrazines ($\text{R}_2\text{N}-\text{N}=\text{S}=\text{O}$) with lithium aluminum hydride yielded highly colored *N*-thionitrosoamines (**95**), which are stable at $< ca. -30^\circ\text{C}$ (66JA3842). However, it was deduced from spectroscopic data that these were not true $\text{RN}=\text{S}$ species; a high contribution from the dipolar resonance form accounts for their stability. Nonetheless, compound **95** will act as a 2π component in an inverse electron demand Diels–Alder reaction with a tetrazine derivative to yield triazole **96** (79CZ230).



Ab initio quantum chemical calculations concluded that $\text{HN}=\text{S}$ is more stable than the isomeric structure $\text{HS}=\text{N}$ and that thermal isomerization between the two species is unlikely (86IC4221; 81PS325). It is now clear, both from experiment and from theory, that electron-donating substituents attached to nitrogen stabilize the $\text{RN}=\text{S}$ system, while electron-withdrawing substituents destabilize these compounds. This is the same effect observed for substituents attached to the carbon of thioaldehydes and selenoaldehydes.

B. PREPARATIVE METHODS AND REACTIONS

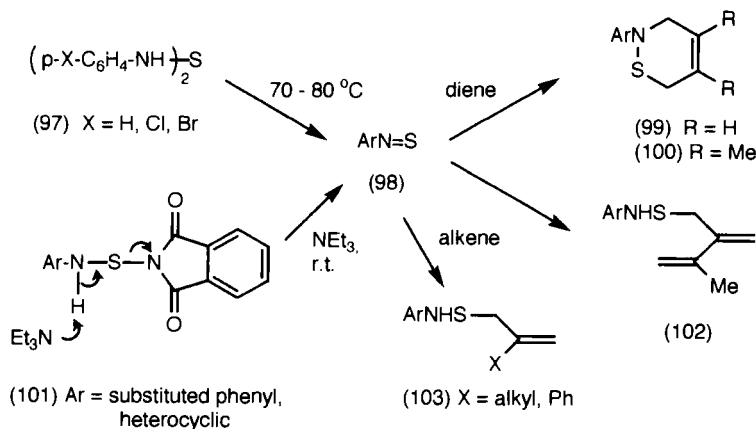
It is likely that thionitroso intermediates are involved in the formation of sulfurdiimides ($\text{RN}=\text{S}=\text{NR}$) by direct reaction of amines with sulfur halides, but this has not proved to be a synthetically useful route to $\text{RN}=\text{S}$ species [78ZC323; 84JCS(P1)2591; 85PS277]. Three routes will be considered:

- (1) Fragmentation of sulfenamides
- (2) Fragmentation of heterocyclic *S,N*-ylides
- (3) Ring opening of 2,1-benzisothiazoles

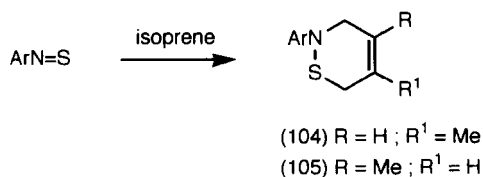
1. Fragmentation of Sulfenamides

Several compounds of general formula $\text{RNH}-\text{S}-\text{X}$ undergo 1,2-elimination of HX , either thermally or upon treatment with base, to generate transient $\text{RN}=\text{S}$ species. This approach has provided substituted arylthio-nitroso derivatives. Tavs first reported that mild thermolysis ($70-80^\circ\text{C}$) of thiodiamines (**97**) in dimethylbutadiene as solvent led to the isolation of *N*-aryl-1,2-thiazines (**100**) (22–41% yield); this reaction provided the first good evidence for the intermediacy and trapping of $\text{ArN}=\text{S}$ (**98**) [66AG(E)1048].

Similar precursors have been investigated briefly by other workers (72JOC3810; 76JOC1333). More recently, *N*-(arylaminothio)phthalimides (**101**) have proved to be readily accessible and versatile $\text{ArN}=\text{S}$ precursors [88CC950; 89TL3835; 90JCS(P1)3225]. A wide range of substituted benzene and heterocyclic (e.g. 3-pyridyl and 2-thiazolyl) derivatives (**101**) fragment at room temperature upon treatment with triethylamine. Trapping of species **98** thereby generated is efficient: the expected adducts **99** are formed with butadiene. With dimethylbutadiene as trap, the results are particularly interesting: -ene adducts **102** are formed in competition with the Diels–Alder adducts **100**. Remarkably, the ratio of products **102**:**100** formed in these reactions is very sensitive to the electronic nature of a substituent on the aryl ring of **98**. Electron-donating substituents favor Diels–Alder reaction, while electron-withdrawing substituents favor -ene reaction. The -ene reaction is regiospecific with $\text{C}-\text{S}$ bond formation [*c.f.* thioaldehyde reactions, which yield both regioisomers, e.g. **56** and **57**]. Alkenes, such as isobutene, α -methylstyrene, α -pinene, β -pinene, and *L*-methylcyclohexene readily form adducts with $\text{ArN}=\text{S}$ of general formula **103** [90JCS(P1)3225; 91UPI].

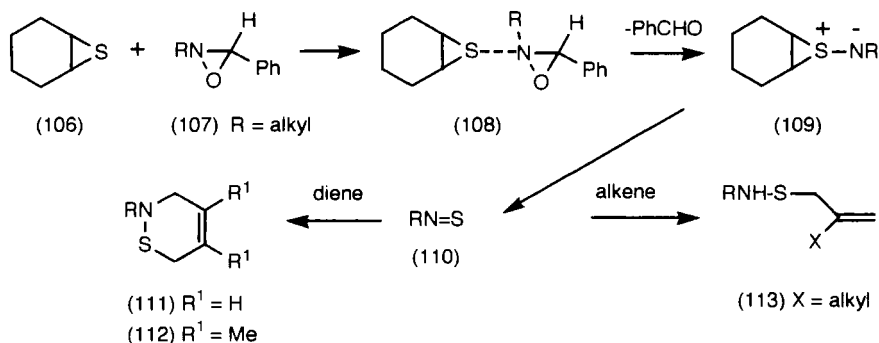


Other dienes that react with $\text{ArN}=\text{S}$ include 1,4-diphenylbutadiene, isoprene, 1-methylbutadiene, chloroprene, (*E,E*)- and (*E,Z*)-hexadienes and 1,1-bicyclohexenyl [90JCS(P1)3225; 91UPI]. Reactions of isoprene with $\text{ArN}=\text{S}$ are regioselective: adducts **104** and **105** form in a 3 : 1 ratio for both 4-methoxyphenyl- and 4-bromophenyl-thionitrosobenzene (88CC950). Addition of (*E,E*) and (*E,Z*)-hexadienes to $\text{ArN}=\text{S}$ occurs with retention of diene stereochemistry [89TL3835; 90JCS(P1)3225]. Russian workers have recently reported that thermal fragmentation of *N*-trimethylsilyl-*N*-chlorothioalkylamines yields alkylthionitroso compounds (90ZOR1799).



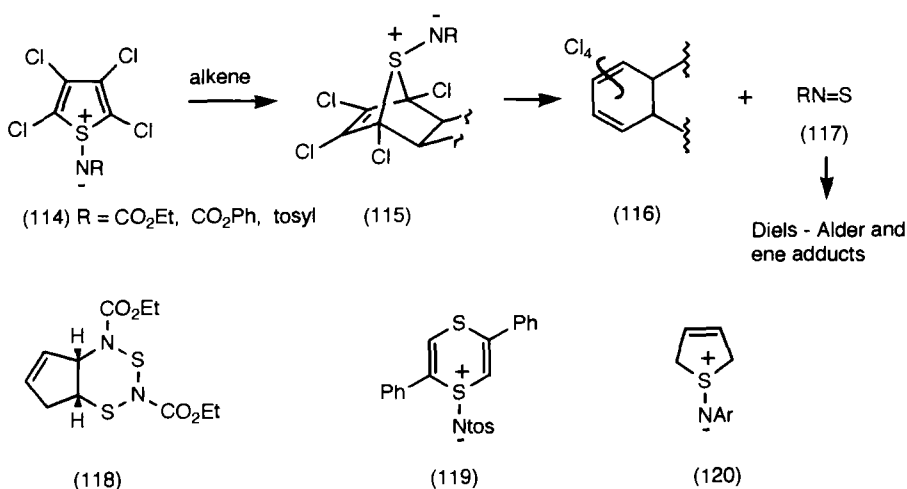
2. Fragmentation of Heterocyclic S,N-Ylides

Hata and Watanabe first used this approach for the formation and trapping of alkylthionitroso compounds **110** (80JOC1691). The key step is fragmentation of the transient ylide (**109**); in the presence of butadiene, adducts **111** are produced, whereas dimethylbutadiene affords **112** as the major product with a small amount of the corresponding -ene adduct (91UPI). Trapping of **110** (generated from *N*-trimethylsilyl-*N*-chlorothioalkylamines) with alkenes, e.g., α -pinene, provides a new route to *N*-alkyl-sulfenamides **113** (91UPI).



Meth-Cohn and van Vuuren have exploited the stable tetrachlorothiophene ylides (**114**) as precursors to highly electron-deficient thionitroso

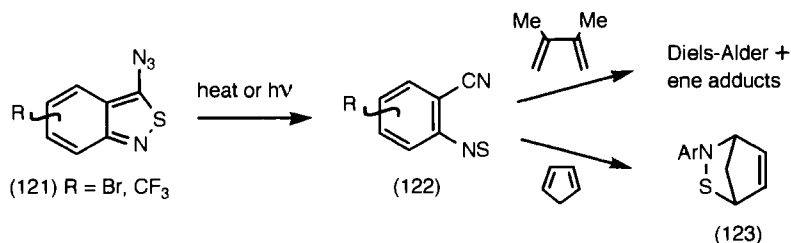
compounds (**115**) [84CC1144; 86JCS(P1)245]. The $\text{RN}=\text{S}$ fragment is expelled as a consequence of cycloaddition of an alkene (usually acenaphthylene) to the diene (**114**). These workers were the first to identify -ene products from thionitroso compounds and to establish that these reactions are regiospecific (like $\text{RN}=\text{S}=\text{O}$, but in contrast to $\text{RCH}=\text{S}$). Indeed -ene reactions of **117** occur very readily due to the electron-withdrawing acyl substituents on nitrogen. The reaction of $\text{EtO}_2\text{C}-\text{N}=\text{S}$ with cyclopentadiene gave the novel heterocycle **118** in low yield.



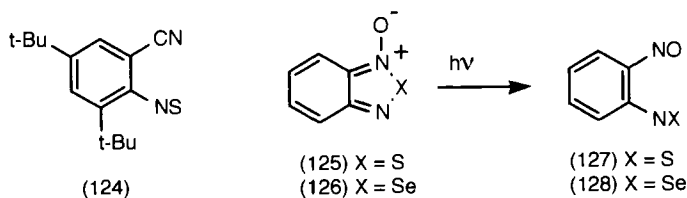
Other ylidic systems, **119** (86MI1) and **120** (91UP1), have been investigated without success as potential $\text{RN}=\text{S}$ precursors.

3. Ring Opening of 2,1-Benzisothiazoles

Joucla and Rees first generated thionitroso compounds in this way: azides (**121**) decompose either photochemically or upon gentle thermolysis to generate the intermediate *o*-cyanothionitrosobenzene derivative **122**, which can be trapped as Diels-Alder adducts by dimethylbutadiene or cyclopentadiene (e.g., bicyclic structure **123** (84CC374). A reinvestigation of the trapping of a range of species (**122**) formed by this method has established that a combination of electronic and steric effects of the cyano substituent ortho to the reactive $\text{N}=\text{S}$ bond greatly facilitates -ene reaction. For example, compound **121** ($\text{R} = 6-\text{Br}$) and dimethylbutadiene at 20°C afford a mixture of -ene and Diels-Alder adducts in the ratio 9 : 1 (91TL7459).



Okazaki and co-workers have recently obtained spectroscopic evidence at low temperatures ($< 90\text{K}$) for the existence of the di-*t*-butyl derivative (**124**) obtained photolytically from the corresponding 2,1-benzisothiazole (89CL2083; 92JA1830). The absorption at $\sim 470\text{ nm}$ assigned to species **124** is in agreement with previous data obtained by Pedersen *et al.* during the photolysis of benzo[*c*]1,2,5-thiadiazole-2-oxide (**125**) at 20K , which was considered to produce the thionitroso species **127** [78ACS(B)625].



VI. Selenonitroso Compounds, $\text{RN}=\text{Se}$

The only evidence for the existence of a selenonitroso intermediate is provided by UV spectra of the photolysate of benzo[*c*]1,2,5-selenadiazole-2-oxide (**126**) in a matrix at 100K . Spectra are consistent with the formation of **128** (79TL745). Telluronitroso compounds, $\text{RN}=\text{Te}$, are unknown.

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The Literature of Heterocyclic Chemistry, Part IV

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I. Introduction

This survey is a sequel to three already published in *Advances in Heterocyclic Chemistry* [66AHC(7)225; 79AHC(25)303; 88AHC(44)269]. It includes monographs and reviews published during the period 1987–1990 as well as some published earlier but omitted in Part III.

Like the previous one, this survey is based mainly on short bibliographic papers published by the authors in *Khimiya Geterotsiklicheskikh Soedinenii* since 1987 (87KGS998, 87KGS1427; 88KGS564, 88KGS1431, 88KGS1572; 89KGS279, 89KGS708, 89KGS1573; 90KGS703; 91KGS420, 91KGS706, 91KGS1282, 91KGS1571). Sources not only in English but also in Russian, German, Japanese, Chinese, French, Czech, Polish, and other languages are surveyed and classified. This feature of the survey should cause no problem because some of the sources are available in English translations and practically all others have informative English abstracts as well as quite understandable and useful schemes and lists of references.

As before carbohydrates are not covered. Such compounds are mentioned only in general cases (e.g., anomeric effect) as well as when carbohydrates serve as starting compounds for the synthesis of other heterocyc-

cles or they are present as fragments of a complex system including another heterocyclic moiety such as nucleosides.

II. General Sources and Topics

A. GENERAL BOOKS AND REVIEWS

1. Textbooks

88MI48.

2. Annual Reports

a. *Comprehensive Reports*. 87AR(B)181; 88AR(B)181; 89AR(B)191.

b. *Specialized Reports*. 86MI20; 89MI14; 90JOM93, 90JOM285.

c. *Synthesis of Saturated Heterocycles*. 86GSM(8)407; 87GSM(9)536; 88GSM(10)457.

3. Other Reviews

a. *General Problems of Heterocyclic Chemistry. Heteroaromaticity*. 86MI8; 89KGS3; 90UK197.

b. *Nomenclature of Inorganic Rings*. 89PS(41)183.

4. History of Heterocyclic Chemistry, Biographies

Personal activities of Sir. D. H. R. Barton: 89H(28)1; H. Böhme: 86MI26; A. V. Bogatskii: 86MI1; E. Campaigne: 90MI66; T. Kametani: 90H(30)1; A. Mangini: 87MI64; C. J. Pedersen: 88AG(E)1021.

5. Bibliography of Monographs and Reviews

a. *Comprehensive Data*. 87KGS998, 87KGS1427; 88AHC(44)269, 88KGS564, 88KGS1431, 88KGS1572; 89KGS279, 89KGS708, 89KGS1573; 90KGS703; 91KGS420, 91KGS706, 91KGS1282, 91KGS1571.

b. *Specialized Surveys*. 86AHC(40)1; 89MI22.

B. GENERAL TOPICS BY REACTION TYPE

1. *Structure and Stereochemistry*

a. *Theoretical Aspects.* 88AG(E)1437, 89MI11.

b. *Molecular Dimensions.* Activation and reaction volumes in solution: 89CRV549.

Crystal and molecular structures: 87MI11; 88MI39.

Molecular structure in the gas phase: 90MI4.

c. *Molecular Spectra.* ESR: 88ACR107.

¹⁷O NMR and assessment of steric perturbation of structure: 89T3613.

One-bond carbon-carbon spin-spin coupling constants: 89MI31.

d. *Stereochemical Aspects.* Anomeric effects: 90H(31)1157.

Conformations of acyl groups in heterocycles: 87AHC(41)75.

Conformations of alkyl and analogous groups: 89APO(25)1.

Conformations of polymethynic dyes possessing azole fragments: 87UK466.

Quantitative study of steric effects in heteroaromatic compounds: 88AHC(43)173.

e. *Betaines and Other Unusual Structures.* Aminonitrenes having heterocyclic structure: 86MI3.

Antiaromatic azacycl/3.3.3/azines: 87H(26)2757.

Cyclic ammonium ylides: 90AKZ649.

Cyclic isoimides and isoimidium salts: 87WCH755.

Heterocyclic analogs of ferrocene and macroheterocycles containing ferrocene fragments: 90JOM93.

Heterocyclic dianions: 88T6957.

Heterocyclic ylides: 79ZVK496.

Mesoionic compounds: 90YGK672.

Pseudoazulenes: 87UK95.

Stable nitroxyl radicals: 79ZVK156; 90MI48.

f. *Miscellaneous Substituted Heterocycles.* Azomethine ylides: 89AHC(45)231.

Boron-substituted heteroaromatic compounds: 89AHC(46)143.

Dithiocarboxyl esters: 88SR(8)155.

Fluorinated heterocycles: 88MI2; 90UK149, 90YGK16.

Heteroaromatic sulfoxides and sulfones: 90AHC(48)1.

Heterocyclic quinones: 89AHC(45)37.
Hetarylsubstituted α,β -unsaturated ketones: 89MI23.
Heterocyclic propellanes and dispiranes: 87MI28.
Schiff bases—heterocyclic derivatives: 89MI24, 90MI24.
Selenoaldehydes—heterocyclic derivatives: 88YGK1149.
Sulfenamides: 89CRV689.
Sulfenylchlorides: 89MI25.
Thioaldehydes: 88YGK1149; 90UK649.

2. Reactivity

a. *General Topics.* Asymmetric induction using heterocycles: 89AHC(45)1.

Asymmetric catalysis using alkaloides: 86TS87.

Basicity and acidity of azoles: 87AHC(41)187.

Carbon-carbon bond introduction in electron-deficient heteroaromatics: 90UK1288.

Counterattack reagents in reactions of heterocycles: 89T1233.

Electrochemistry of heterocycles: 87MI10.

Electron transfer in reactions of heterocycles: 89MI12.

Electronic effects of heteroaromatic and substituted heteroaromatic groups: 87AHC(42)1.

Enzymes in transformations of heterocycles: 86T3351; 90MI63, 90S1, 90T6587.

Expert system for prediction of reactions: 87MI29.

Gas-phase reactions of heteroaromatics: 86AHC(40)25.

Gas-phase reactions of heterocyclic anions: 88APO(24)1.

Heterocycles as ligands: 87MI47.

Heterocycles as umpolung synthons: 87MI39.

Heterocyclic quinoneimines, reactivity of: 89KGS1011.

High-pressure reactions of heterocycles: 89YGK321.

Insertion reactions of heterocycles with isocyanates: 87ZC77.

Investigation of reactions of heterocycles by the use of radioactive sulfur: 88MI18; 90MI14.

Lanthanides in reactions of heterocycles: 86T6573.

Laser-induced reactions of heterocycles: 90YGK536.

Nickel complexes in transformations of heterocycles: 90YGK370.

Palladium salts and complexes in reactions of heterocycles: 90S739.

Quinones and quinone methides in chemical modification of heterocyclic fragments in biopolymers: 89AG(E)555.

Reaction mechanisms of heterocycles: 89MI4.

Ring opening in five-membered heteroaromatic anions: 87AHC(41)41.

Ring-opening polymerization of heterobicyclic compounds: 89YGK1040.

Solid-phase reactions of heterocycles: 89BSF237.

Sonochemistry of heterocycles: 89H(29)597, 89S787.

Transition metals in transformations of heterocycles: 90JOM285.

b. *Reactions with Electrophiles and Oxidants.* Aminomethylation of heterocycles: 90T1791.

Dimethylsulfoxonium methylide, reactions with heterocycles: 87T2609.

Electron-transfer mechanism in electrophilic nitration of activated heteroaromatic compounds: 87ACR53; 88UK254.

Electrophilic amination of heterocyclic carbanions: 89CRV1947.

Electrophilic substitution of heterocycles, quantitative aspects: 90AHC(47)1.

Fluorination of heterocycles: 87MI16; 88OR(35)513.

Formylation of heterocycles with acetic formic anhydride: 90T1081.

Oxidation of heterocycles: 87AHC(41)275; 88MI43, 88MI52, 88OR(35)421; 90WCH111.

Proton transfer by heterocyclic free radicals: 88UK1440.

Reactions of heterocycles with aliphatic nitrocompounds: 88CSR283.

Reactions of heterocycles with hydroxy(organosulfonyloxy)-iodo/ar-
enes: 90MI57.

Reactions of heterocyclic free radicals with organic cations: 88UK50.

c. *Reactions with Nucleophiles and Reducing Agents.* Amination of heterocycles (Chichibabin reaction): 88AHC(44)1.

Heterocyclic cations, reactions with aliphatic diazocompounds: 85MI3.

Hydroboration of heterocycles: 90JHC13.

Metallation of heterocycles: 87MI24, 87MI36; 88MI14; 90CRV879, 90MI10.

Nucleophilic ring opening of saturated heterocycles: 88KGS1155.

Reactions of heterocycles with sulfur-containing dianions: 88Y21.

Reduction of heterocycles: 86MI15; 89CRV459.

Transition metal activated nucleophilic substitution in heteroaromatic compounds: 90BSF401.

Vicarious nucleophilic substitution of hydrogen in heterocycles: 88MI35; 89UK1298.

Wittig olefination in heterocycles: 89CRV863.

d. *Reactions toward Free Radicals, Carbenes, etc.* Alkylation of heteroaromatic bases with α -hydroxyalkyl radicals: 90KGS579.

Amino- and hydroxynitrenes, reactions with heterocycles: 89UK1271.

Carbene complexes of heterocycles: 79ZVK505; 87MI30; 89AG(E)397.

Cobalt-mediated radical reactions of heterocycles: 88CSR361.

Free radical chain reactions of heterocycles: 88S417, 88S489.

Radical reactions of hetarenediazonium ions; 88CRV765.

Radical reactions of heterocycles, general reviews: 86MI14; 87T3541.

Radical reactions of heterocyclic thiocarbonyl derivatives: 88MI11; 89CRV1413; 90MI7.

Reactions of heterocyclic cations with free radicals: 88UK50.

Regioselectivity in radical addition and substitution of heterocycles: 88MI7.

Stereoselectivity of intermolecular free radical reactions of heterocycles: 89AG(E)969.

Substitution reactions of heteroaromatic bases with nucleophilic free radicals: 89H(28)489; 90JHC79.

e. *Heterogeneous Catalysis.* Conversion of heterocycles on oxide catalysts: 87MI9.

Vanadium catalysts for oxidation of heterocyclic compounds: 90MI3.

f. *Reactions with Cyclic Transition State.* Hetero-Cope rearrangement: 89S71.

Heterocyclic dienes and heterocyclic olefins in [4 + 2]- and [2 + 2]-cycloaddition reactions: 88T6755.

Heterocycles, reactions with tetracyanoethylene: 87S749, 87WCH21.

g. *Reactivity of Substituents.* Brominated heteroaromatic compounds, reactivity: 88MI50.

Claisen rearrangement in heteroaromatic systems: 87AHC(42)203.

Control of carbon versus oxygen acylation of enolate anions of heterocyclic carbonyl derivatives: 89OPP179.

Electrochemistry of azomethines derived from heterocycles: 89MI13.

Heteroaromatic sulfoxides and sulfones, exchange and coupling of ligands: 90AHC(48)1.

Oxidation of alcohols derived from heterocycles: 90S857.

Heterocycles, palladium-catalyzed carbonylation: 88UK529.

Pummerer reaction of heterocyclic compounds: 89KGS1299.

Wittig olefination of carbonyl derivatives other than aldehydes and ketones: 88CSR1.

h. *Heterocycles as Intermediates in Organic Synthesis.* N-Acylated heterocycles and cyclic derivatives of carboxylic acids in the synthesis of aldehydes: 890PP451.

- Azoles as intermediates in organic synthesis: 87S857; 88MI57.
Cyclic acetals and dithioacetals as protecting groups: 89OPP705.
Cyclic acetals of chiral diols in asymmetric synthesis: 87YGK944.
Cyclic nitrosonium salts as oxidants: 88H(27)509.
Cyclic vinyl acetals in organic synthesis: 89S721.
Heterocycles in asymmetric synthesis of α -amino acids: 87UK1832.
Heterocycles in organic synthesis, general problems: 90AG(E)1320, 90JHC31.
Heterocycles in synthesis of β -fluorinated amino acids: 90UK149.
Heterocycles in synthesis of 4-oxocarbonyl compounds: 89OPP659.
Heterocyclic o-chloroaldehydes as synthons: 89PS(43)289.
Heterocyclic oxochromiumamine complexes as oxidants: 88OPP533.
Heterocyclic S,S-, N,S-, and N,N- α -oxoketene acetals as intermediates in organic synthesis: 90T5423.
Heterocyclic protecting groups for the amino group: 89CRV149.
Heterocyclic protecting groups for the carboxy group: 84MI12; 89CLY803.
Heterocyclic protecting groups for the mercapto group: 89CLY463.
Heteroquadricyclanes in organic synthesis: 89CRV1203.
Nickelocycles in organic synthesis: 88AG(E)186.
Organomercury derivatives of heterocycles in synthesis: 85MI12.
Organosilicon and organotin derivatives of thiazole and oxazole as intermediates in organic synthesis: 88G211.
Oxaziridines in organic synthesis: 89T5703.
Stereocontrolled cyclofunctionalizations of double bonds via heterocyclic intermediates: 90T3321.
3-Sulfolenes as precursors of 1,3-dienes: 88YGK893; 89OPP257.
Thiocarbonyl heterocyclic derivatives in organic synthesis: 87MI33.

3. *Synthesis*

a. *General Topics* Aliphatic diazocompounds as carbene precursors and carbene synthesis of heterocycles: 85MI1, 85MI2, 85MI6, 85MI7; 87MI30; 89UK1233, 89UK1250.

Allyl 1,3-strain and formation of heterocycles from heteroallyl systems: 89CRV1841.

Aminoacetals in synthesis of heterocycles: 87H(25)601.

Amino acids in asymmetric synthesis of heterocycles: 87MI25.

Azides in synthesis of heterocycles: 88CRV297.

Computer-assisted mechanistic evaluation of heterocyclization reactions: 88JOC2504.

Counterattack reagents in synthesis of heterocycles: 89T1233.

Cyclic enamines, ynamines and ureas, preparative methods: 86MI12.

Design of free radical reactions for the synthesis of heterocycles: 88S417, 88S489.

Effect of heteroatoms on the synthesis of heterocycles by intramolecular cyclization: 87YGK992.

Enantioselective syntheses of heterocycles: 89CRV1663; 90ACR207.

Enzymatic syntheses of heterocycles: 90MI63, 90T6587.

Expert system for synthetic design of heterocycles: 87MI29.

Isothiocyanates in the synthesis of heterocycles: 89MI36.

Lanthanides in heterocyclic synthesis: 86T6573.

Metallation in synthesis of heterocycles: 87MI31, 87MI36.

Mechanisms of heterocyclization: 87T5171.

Nitrenes in heterocyclic syntheses: 86MI3; 87ACR18; 89UK1271; 90KGS291.

Nitriles in synthesis of heterocycles: 87H(26)497.

Pericyclic reactions in the synthesis of heterocycles: 87YGK60.

Pummerer reaction in the synthesis of heterocycles: 89KGS1299.

Retro-Diels-Alder strategy in the synthesis of heterocycles: 87S207.

Solid-phase synthesis of heterocycles: 89BSF237.

Sulfonium ylides in heterocyclic synthesis: 90KGS127.

Synthesis of heterocycles at high pressure: 89YGK321.

Thioamide groups in heterocyclization: 87YGK682; 88H(27)1953.

Transition metals in heterocyclic synthesis: 87YGK244; 88CSR361, 88H(27)2225, 88MI24; 89AG(E)1173, 89CRV1663, 89CRV1927, 89MI7, 89T6901; 90BSF401, 90JOM285, 90S739.

Umpolung synthons in synthesis of heterocycles: 87MI39.

b. *Ring Synthesis from Nonheterocyclic Compounds.* Acetoacetanilides in synthesis of heterocycles: 88JHC9.

Acetylenic carbonyl compounds in synthesis of heterocycles using reactions with dinucleophilic reagents: 87KGS291.

Acetylenic ethers and their analogs in synthesis of heterocycles: 89UK1671.

3-Alkoxyacroleins in heterocyclic synthesis: 87S1.

3-Alkoxypropenic acid derivatives in synthesis of heterocycles: 88MI31.

Amidrazones in synthesis of heterocycles: 89KGS867.

α -Aminonitriles in synthesis of heterocycles: 89UK250.

3-Aminothioacrylamides in heterocyclic synthesis: 87ZC8.

Asymmetric synthesis of heterocycles from olefins via cyclization with the formation of carbon-heteroatom bonds: 84MI1.

Azomethines, electrochemical heterocyclization: 89MI13.

Azomethineimines, cycloaddition reactions: 86MI5.

Bifunctional hydroxylamine derivatives as precursors of heterocycles: 87MI49.

Carbon disulfide in synthesis of heterocycles: 89JHC1167.

Chlorocarbonyl isocyanate in synthesis of heterocycles: 90H(31)1377.

Chlorosulfonyl isocyanate in heterocyclic synthesis: 87H(26)1051.

α -Cyanothioacetamide in synthesis of heterocycles: 87H(26)205.

Cycloaddition reactions of α -halonitroso compounds: 87OPP329.

Cycloalkylation of substituted aldehydes and ketones with the formation of heterocycles: 87CRV1277.

Cyclohexane-1,3-diones in synthesis of heterocycles: 88KGS723.

Developments in heterocyclization reactions: 90T1385.

N,N-Dialkyl-1,3-diene-1-amines, cyclization: 90RTC311.

Diels-Alder cycloaddition reactions in synthesis of natural heterocycles: 87AHC(42)245.

1,5-Diketones, heterocyclization under catalytic reduction conditions: 90KGS1011.

1,3-Dipolar cycloaddition, monograph: 87MI55.

Dithioketene acetals in synthesis of heterocycles: 90MI65.

Free radical heterocyclization of unsaturated compounds using carbonyl compounds in the presence of Mn(III) acetate: 89UK475.

Functionalized nitroalkanes in synthesis of heterocycles: 88S833.

Hydrogen cyanide derivatives in synthesis of heterocycles: 87AHC(41)1.

Intramolecular addition of an amino group to a nitrile group: 87MI1.

Intramolecular addition of OH, NH, COOH to an acetylene moiety: 87YGK112.

Intramolecular aryne arylation in synthesis of heterocycles: 89ACR275.

Nitrile oxides, cycloaddition reactions: 90H(30)719.

Nitrile oxides, nitrones, and nitronates in synthesis of azoles: 88MI57.

Nitrones, cycloaddition reactions: 86MI17; 88OR(36)1.

Nitrile sulfides in synthesis of heterocycles: 89CSR33.

Nitroalkenes in heterocyclic syntheses using 1,3-dipolar cycloaddition: 90KGS435.

Nucleophilic ring opening and formation reactions of saturated heterocycles: 88KGS1155.

Oxidative heterocyclization using hypervalent iodine: 90S431.

Phosphoroamides in synthesis of heterocycles: 87WCH513.

N-Protected optically active α -amino aldehydes in synthesis of heterocycles: 89CRV149.

Prototropic routes to 1,3- and 1,5-dipoles, and 1,2-ylides as intermediates in synthesis of heterocycles: 87CSR89.

Tetracyanoethylene in synthesis of heterocycles: 87WCH21.

α -Thiocarbocations as intermediates in synthesis of heterocycles: 89YGK330.

Vinylisothiocyanates in synthesis of heterocycles: 89ZC41.

c. *Syntheses by Transformation of Heterocycles.* Acylation of enolate anions in synthesis of heterocycles: 89OPP179.

Bihetaryl compounds, synthesis of: 90AG(E)977.

Brominated heteroaromatic compounds: 88MI50.

Chiral heterocycles in asymmetric synthesis of heterocycles: 84MI15.

Fluorinated heterocycles: 89YGK619; 90YGK16.

Heterocyclic haloimidoyl compounds: 87UK1973.

Hetarylisocyanates: 87MI13.

Hydroboration in synthesis of racemic and optically active heterocycles: 87H(25)641; 88ACR287.

Mesoionic compounds as building blocks of heterocycles: 90YGK672.

Organomercury derivatives of heterocycles: 85MI12.

Organoselenium derivatives of heterocycles: 86MI16.

4. *Properties and Applications (Except Drugs and Pesticides)*

a. *Dyes and Intermediates.* Dyes with absorption in near IR region: 89MI28.

Polymethyne dyes: 87UK466; 89MI9.

Pyrazolone couplers for color photography: 87YGK151.

Syntheses, properties, and applications, general monograph: 87MI56.

b. *Substances with Luminescent and Related Properties.* Chemi- and bioluminescence of heterocycles: 87MI26.

Luminescent spectroscopy and photochemistry of pyridinium salts: 87H(26)2963.

Photochromism and other phototropic properties: 87YGK837; 88CRV183, 88MI34; 89AG(E)413; 90MI34, 90MI35, 90UK1144.

c. *Organic Conductors (Except Polymers).* Dithiolate complexes: 90UK1179.

Fused heterocycles: 88YGK955; 89YGK1108.

Phthalocyanines with photoconductive and electrochromic properties: 87YGK837.

Structurally stable ensembles of metallomacroheterocycles: 90AG(E)857.

Tetrachalcogenofulvalenes and their charge-transfer complexes: 87MI61, 87YGK502.

d. *Coordination Compounds*. Analytic reagents: 88MI4.

Chelates and coordination compounds with heterocyclic ligands, general problems: 87MI47, 87MI57.

Metallocomplexes of heterocycles as catalysts: 87CRV1401, 87UK754; 89YGK1017; 90UK1960.

e. *Polymers*. Catalytic methods for syntheses of polybenzazoles: 89MI32.

Chemical modification of polymers with the use of heterocycles: 90MI53, 90MI54.

Cross-coupling reactions based on cyclic acetals: 87S1043.

Electrochemical synthesis and properties of polypyrrole: 87WCH239.

Filmforming electropolymerization of heterocycles: 87CLY673.

Functionalized polypyrroles as materials for electrocatalysis and related applications: 89ACR249.

Hexafluoroisopropylidene-substituted polyheteroarylenes: 87UK489.

Nucleophilic aromatic nitrosubstitution in the synthesis of polyimides: 88MI65

Optical properties of conducting polymers containing heterocyclic fragments: 88CRV183.

Perspectives on the application of functional liquid-crystalline polymers and composites including those containing heterocyclic fragments: 90MI36.

Photosensitive polymers having heterocyclic fragments: 90MI34.

Polymers based on epoxide oligomers and polyheteroarylenes: 90MI33.

Polymers containing heterocyclic fragments as matrixes for composites: 90MI32.

Polymers containing heterocyclic fragments as organic ferromagnetics: 90UK529.

Polymers containing N-heterocyclic fragments: 87BSF696.

Polymers containing O-heterocyclic fragments and polymerization of O-heterocycles: 90IZV2321.

Polymer-supported reagents and catalysts for acylation: 89MI30.

Polytriazines: 87MI17; 89UK1528.

Potential conducting material poly(1,2-dithiolo-1,2-dithiol-2,6-diylidene): 89PS(43)165.

Pyrolysis of polyimides: 88UK1742.

Thermal polymerization and oligomerization of heterocyclic monomers: 87UK865.

Thermostable polymers containing heterocyclic fragments: 90AG(E)1262.

f. *Miscellaneous.* Heterocycles as organic ferromagnetics: 90UK529.
Heterocycles as photoaffinity reagents for the analysis of protein structure: 88YGK1041.

Heterocycles as solvents: 86MI21.

Heterylisocyanates, physicochemical properties: 87MI3.

Stable heterocyclic nitroxyl radicals as reagents for investigation of homolytic processes: 87UK1253; 88UK1440.

C. SPECIALIZED HETEROCYCLES

1. *Nitrogen Heterocycles (Except Alkaloids)*

a. *General Sources and Topics.* Acid-base properties of N-heterocycles: 85MI14; 87AHC(41)187; 90MI20.

Antiaromatic azacycl[3.3.3]azines: 87H(26)2757.

Aromatic and antiaromatic nitrogen heterocycles, comparison of quantum-chemical methods for: 89KGS1587.

Azaadamantanes with bridgehead nitrogen atoms: 89UK1815.

Aza-Cope rearrangement in the synthesis and transformations of N-heterocycles: 87UK814.

N-Carboxylic acids of nitrogen heterocycles: 87H(26)1333.

Chiral N-heterocycles as ligands for asymmetric catalysis: 85MI0.

Chiral N-heterocycles with chiral nitrogen centers, synthesis, and applications: 84MI17.

Cyclic sulfenamides: 89CRV689.

Cyclic sulfonamides: 88AHC(44)81.

N-Dithiocarboxylic acids of nitrogen heterocycles: 87H(26)1657.

Intrabridgehead interactions and reactions in diazabicyclics: 90T682.

Lactam acetals: 88T5975.

Nitrogen heterocycles in petroleum: 88MI1.

"Proton sponges"—heteroaromatic bases with exceptional basicities: 88AG(E)865.

Ring-chain tautomerism of functionally substituted hydrazones: 88KGS3.

Stable heterocyclic nitroxides: 79ZVK156; 90MI46.

Strain effects on basicities of N-heterocycles: 89CRV1215.

b. *Structure and Stereochemistry.* Conformations of saturated nitrogen mono- and biheterocycles: 88MI44.

EPR spectroscopy of N-heterocyclic radicals: 88ACR107; 90MI64.

N-Hetarenechromiumtricarbonyls: 87UK1190.

N,S- and B,N,O-Heterocycles, molecular structure in the gas phase: 90MI4.

Mass spectrometry of biologically active N-heterocycles: 87MI4.

^{15}N NMR study of N-heterocycles: 89T581.

Radical cations derived from N- and N,S-heterocycles, generation and structure investigation: 90MI51.

"Proton sponges" derived from N-heteroaromatic bases, geometry of hydrogen bonds: 99AG(E)865.

X-Ray fluorescence spectroscopy of N-arylthiosubstituted derivatives of saturated N-heterocycles: 88MI21; 90MI17.

Strain effects on basicities of saturated N-heterocycles: 89CRV1215.

c. *Reactivity.* Activation and reaction volumes of N-heterocycles in solution: 89CRV549.

N-Alkoxy lactams in preparative organic chemistry: 90YGK749.

Amination of N-heterocycles (Chichibabin reaction): 87KGS1011; 88AHC(44)1; 90AHC(49)117.

Catalytic hydrodenitrogenation of N-heterocycles: 88MI58.

Electrochemical generation of N-anions from lactams: 89YGK939.

Fluorination of N-heterocycles: 88OR(35)513; 90CLY952.

Free-radical substitution of N-heteroaromatic compounds: 89H(28)489; 90JHC79.

Heterocyclic aminyl radicals as free radical inhibitors: 87CRV1313.

Heterocyclic enamines in asymmetric synthesis: 86MI10.

Heterocyclic Mannich bases, reactivity: 90T1791.

Heterocyclic tertiary amines as catalysts for the reaction of activated vinyl carbanions with aldehydes: 88T4653.

Hydride shifts and transfers in N-heterocycles: 88APO(24)57.

Oxidative transformations of heteroaromatic iminium salts: 87AHC(41)275.

Pharmacologically active N-heterocycles, synthetic approach to: 87YZ459.

Photochemical reactions of N-heterocyclic radicals in the solid phase: 87MI51.

Reduction of N-heterocycles with complex metal hydrides: 86AHC(39)1; 88OR(36)249.

Saturated N-heterocycles, activation of $\alpha\text{-sp}^3$ centers toward electrophilic substitution: 88PIA187.

Saturated N-heterocycles as donors of hydride ions: 89UK2011.

Saturated N-heterocycles, dealkylation with acyl chlorides: 89S1.

C-Substitution of N-heterocycles: 88H(27)2659.

d. *Synthesis*. 3-Alkoxyacroleins in synthesis of N-heterocycles: 87S1.
3-Aminothioacryl amides as synthons for N- and N,S-heterocycles: 88PS(35)5.

Azaadamantanes, synthesis via bridgehead and bridge imines: 87H(26)3265.

Azadienes, cycloaddition reactions: 87H(26)777.

Beckmann reactions (rearrangement and rearrangement-cyclization) in synthesis of N-heterocycles: 88OR(35)1.

Bifunctional hydroxylamine derivatives in the synthesis of N-oxides of N-heterocycles: 87MI49.

Carbenes, reactions with azocompounds: 87UK1324.

3-Chloro-2-aza-2-propeniminium units as building blocks in synthesis of N-heterocycles: 88S655.

α -Cyanothioacetamide in synthesis of N-heterocycles: 87H(26)205.

[2.2.3]Cyclazines and aza[2.2.3]cyclazines, syntheses of: 88H(27)2251.

Diamines in synthesis of N-heterocycles: 89ZC276.

1,3-Dipolar cycloaddition in synthesis of N-heterocycles: 85MI5; 87YGGK269; 88H(27)981.

Enamides in synthesis of N-heterocycles: 87S421.

Hydrazinoacetic acids in synthesis of N-heterocycles: 90PHA1.

Intramolecular amino-nitrile cyclization in synthesis of aminopyrroles, -azoles, -azines, and -azepines: 87MI1.

Ketenimine complexes from carbene complexes and isocyanides as building blocks for N-heterocycles: 88AG(E)1456.

Nitrenes in synthesis of N-heterocycles: 86MI3; 87UK1324; 90KGS291.

N-Nitrosamines in synthesis of N-heterocycles: 87OPP83.

Palladium chelates in synthesis of N-heterocycles: 90RTC567.

Reductive amination in synthesis of N-heterocycles: 87KGS435.

Reductive desulfurization in the synthesis of cyclic ureides and lactams: 88MI17; 90MI13.

N-Vinyliminophosphoranes in synthesis of N-heterocycles: 90YGGK681.

2. Oxygen Heterocycles

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b. *Structure and Stereochemistry*. Cyclic acetals, quantum chemical studies of electron structure and reactivity: 89MI29.

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α -Cyanothioacetamide in the synthesis of S-heterocycles: 87H(26)205.

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Thiocarboxylic anhydrides in the synthesis of S-heterocycles: 87ZC90.

Thioketenes in the synthesis of S- and N,S-heterocycles: 88T1827.

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D. NATURAL AND SYNTHETIC BIOLOGICALLY ACTIVE HETEROCYCLES

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Progress in the chemistry of organic natural products: 87FOR(50), 87FOR(51), 87FOR(52); 88FOR(53), 88FOR(54).

Progress in the total synthesis of natural compounds, annual reports: 86GSM(8)497; 87GSM(9)633; 88GSM(10)550.

Individual total syntheses of heterocycles or via heterocycles: 84MI24, 84MI26, 84MI27, 84MI30.

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Chemistry and biological action of alkaloids: 86MI7; 88MI49.

b. *Structure*. Molecular structures of quinine derivatives. 88MI40.

X-Ray crystal structures of C₁₉-diterpenoid alkaloids: 87H(26)2503.

c. *Synthesis*. Addition of stabilized C-nucleophiles to N-alkylpyridinium salts in alkaloid synthesis: 88H(27)789.

Anodic oxidation in the synthesis of piperidine alkaloids: 90Y GK814.

Antitumor sesbanimides, total synthesis of: 87Y GK983.

1-Azaspiro[5.5]undecane system of histrionicotoxines, syntheses of: 89BSF370.

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Macrocyclic pyrrolizidine alkaloids, total synthesis of: 88Y GK693.

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- Alkaloids of *Licopodium*, synthesis and biological role: 90JHC97.
Alkaloids from blue seaweeds, pharmacological activity of: 88PHA809.
Aporphyne alkaloids of *Liriodendron tulipifera*: 87KPS628.
Dimeric quinoline alkaloids: 89KPS4.
4,5-Epoxydihydromorphinones in the synthesis of opioid receptor antagonists: 89YGK374.
Isoquinoline alkaloids, analysis of: 89CLY716.
Morphinandienone alkaloids: 88H(27)1269.
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Quaternary benzo[c]phenanthridine alkaloids: 90CCC2840.
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- e. *Indole Alkaloids*. Alkaloids *Gelsemium*, syntheses of: 90YGK876.
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Monoterpenoid indole alkaloids, synthesis of: 88YZ461.

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Structure elucidation of antibiotics and preparation of new substances using biosynthetic methods: 88YGK490.
- b. *Antitumor Antibiotics*. Bicyclomycin: 88CRV511.
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Quinone antitumor antibiotics, synthetic studies of: 88YGK801.
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- c. *β -Lactam Antibiotics*. Biosynthesis of penicillins and cephalosporins: 90JHC71.

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Chiral β -lactam antibiotics, S-heterocycles in the syntheses of: 87MI32.

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β -Lactam antibiotics, mechanisms of reactions: 87APO(23)165.

β -Lactam antibiotics, new syntheses using S-ylide rearrangement: 89YZ345.

1-Methylcarbapenem, synthesis of key intermediates for: 89YGK606.

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Vitamin B₁₂, biological role of: 88BSF187.

Vitamin B₁₂, biosynthesis of: 89H(28)1193; 90ACR308.

Vitamin B₁₂, origin of its molecular structure: 88AG(E)6.

Vitamin E and related compounds as antioxidants: 89YGK902.

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Immunotropic activity of azole derivatives: 90KFZ(10)19.

Inhibitors of phosphodiesterase as cardiotonics: 90KFZ(12)13.

Tumor diagnostic and therapy using radiolabeled macrocycle-antibody conjugates: 90CSR271.

c. *Individual Substances and Groups of Compounds.* 2-(2-Amino-thiazol-4-yl)-2-hydroximinoacetic acid derivatives: 88MI64.

Barbituric acid derivatives, history and applications: 88PHA827.

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1,4-Dihydropyridines: 90KFZ(6)14.

1,2-Dithiin derivatives: 89PS(43)209.

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Praziquantel (hexahydropyrazino[2,1-*a*]isoquinoline derivative) as anthelmintic: 90KFZ(9)60.

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Triazine herbicides, metabolism, degradation, and toxicity: 87WCH55.

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b. *Enzymes, Coenzymes, and Their Models*. O(6)-Alkylguanine DNA alkyltransferase, role in anti-tumor activity of N-nitrosoureas: 89KFZ389.

Biopterin cofactor and related compounds, chemistry and biological function of: 88YGK564.

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Enzymes altered at their active sites, catalytic activity of: 88AG(E)913.

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Synthetic DNA molecules as enzyme substrates: 90CRV1327.

Tromboxane- A_2 synthase inhibitors: 87YGK1.

c. *Amino Acids and Peptides*. Muramylpeptides and lipopeptides, studies toward immunostimulants: 89T6331.

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d. *Plant Metabolites*. Cembranes and cembranolides, synthesis of: 88CRV719.

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Plant hormones: 88YGK436.

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Tannins: 90H(30)1185, 90KPS293.

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Crustacean hormones on preparation for molting: 88YGK447.
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f. *Pheromones and Other Substances from Insects*. Insect juvenile hormones and their bioanalogues: 89CCC2303.

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Maillard reaction in food and in human organism: 90AG(E)565.

Maltol as food additive: 90CLY404.

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Immunine regulatory agents, design and synthesis of: 89T4327.

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III. Three-Membered Rings

A. GENERAL TOPICS

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Saturated three-membered heterocycles, addition reactions leading to 5-membered 1,3-heterocycles: 86MI4.

Thermal and photochemical electrocyclic reactions in the chemistry of three-membered heterocycles: 89KGS1443.

Three-membered heterocycles in the synthesis of crown compounds and cryptands: 89KGS1299.

B. ONE HETEROATOM

1. *One Nitrogen Atom*

Alkoxyaziridines, structure, properties, formation from O-nitrenes: 90KGS291.

Aziridine-2-carboxylic acid, asymmetric derivatives of: 87MI5.

Aziridines, formation from nitrenes and unsaturated compounds: 79ZVK485.

Aziridines in the synthesis of azomethine ylides: 89AHC(45)231.

Aziridines in the synthesis of natural products: 86AHC(39)181.

Azirines as intermediates in pyrrole formation: 87KGS1299.

3-Amino-2-*H*-azirines, synthesis and properties: 90KGS867.

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a. *Oxiranes, General Topics.* Radiation chemistry of epoxides: 89MI8.

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b. *Reactivity of Oxiranes.* S_N2' Additions of organocopper reagents to vinyloxiranes: 89CRV1503.

Bicyclic oxiranes in the synthesis of prostanoid synthons: 87MI21.

Cycloaddition reactions of oxiranes: 89YGK102.

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α,β -Epoxysulfoxides in organic synthesis: 89YGK734.

Heterolytic cleavage of oxiranes with Grignard reagents: 89UK401.

Stereospecific deoxygenation of epoxides to olefins: 87H(26)1345.

c. *Synthesis of Oxiranes.* Asymmetric epoxidation with titanium-tartrate catalysts, mechanism of: 85MI11.

Asymmetric epoxidation, synthetic aspects of: 85MI10; 87YGK90.

Epoxidation with hydrogen peroxide catalyzed by heteropoly acids: 89YGK889.

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Syntheses of epoxides involving organoselenium intermediates: 89H(28)1203.

Transition-metal-catalyzed epoxidations: 89CRV431.

3. *One Sulfur Atom*

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Thiiranium ions as intermediates: 88MI15; 90MI11.

C. TWO HETEROATOMS

1. *Two Nitrogen Atoms*

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2. *One Nitrogen and One Oxygen Atom*

Optically active sulfamyloxaziridines in enantioselective oxidation of nonfunctionalized substrates: 87MI35.

Oxaziridines in organic synthesis: 89T5703.

3. *Two Oxygen Atoms*

Dioxiranes as powerful oxidants: 89ACR205.

Dioxiranes, general review: 89CRV1187.

IV. Four-Membered Rings

A. ONE HETEROATOM

1. *One Nitrogen Atom*

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Conversion of β -lactams into versatile synthons via molecular rearrangement and lactam cleavage: 88H(27)1755.

Electrochemical transformations of β -lactams: 90YZ463.

Ester-enolate-imine condensation in the synthesis of β -lactams: 89CRV1447, 89H(29)2225.

Organometallic reagents in β -lactam chemistry: 88T5615.

Stereochemistry of electrophilic substitution in β -lactams: 90KGS1155.

β -Lactam antibiotics (see Section II,D,3,c).

2. *One Oxygen Atom*

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Cycloaddition reactions of oxetanes: 89YGK102.

B. TWO HETEROATOMS

1. *Two Nitrogen Atoms*

Synthesis and reactions of 3-oxo-1,2-diazetidinium ylides: 84MI4.

2. *Two Oxygen Atoms*

Chemistry and chemiluminescence of 1,2-dioxetanes: 90MI25.

3. *One Oxygen and One Sulfur Atom*

β -Sultones: 87T1027.

4. *Two Sulfur Atoms*

Mono-, di-, and tetra-S-oxides of 1,3-dithietanes: 88S349.

V. Five-Membered Rings

A. GENERAL TOPICS

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Electrochemistry of azoles: 90MI27.

Ring-opening of 5-membered heteroaromatic anions: 87AHC(41)41.

B. ONE HETEROATOM

1. *General*

Filmforming electropolymerization of 5-membered heterocycles: 87-CLY673.

Generation of enols in solutions of 5-membered heterocycles and their benzannelated systems: 88ACR135.

2. *One Nitrogen Atom*

a. *Monocyclic Pyrroles*. Electrochemical synthesis and properties of polypyrrole: 87WCH239.

Functionalized polypyrroles as materials for electrocatalysis and related applications: 89ACR249.

Nitropyrroles: 86MI11.

Perspectives of pyrrole chemistry: 89UK1703.

Phototropic pyrrole derivatives: 88MI34.

Synthesis of pyrroles: 87KGS1299, 87MI22; 89KGS291, 89UK275; 90H(31)383.

b. *Hydropyrroles*. (R)- and (S)-Amino-2-methoxymethylpyrrolidines in asymmetric synthesis: 84MI13.

Chiral pyrrolidine derivatives as chiral auxiliaries: 90YGK982.

2,5-Disubstituted pyrrolidines as chiral auxiliaries: 90YGK984.

Nucleophilic addition to maleimides: 87AKZ296.

Proline-catalyzed enantioselective aldol reaction, mechanism of: 88BSF499.

Proline in asymmetric synthesis: 87MI25.

3-Pyrrolidinol, synthesis of: 87H(26)2247.

4-Substituted 2-carboxy-3-pyrrolidineacetic acids (kainoids), syntheses of: 89YGK212.

c. *Pyrrole Pigments*. Biliverdins and bilirubins, chromatographic analysis and structure determination of: 89MI34.

1-Bromo-19-methylbiladienes-ac as precursors to porphyrins: 90MI56.

Microbiologic synthesis of bile pigments: 89MI6.

Linear oligopyrroles and bile pigments, chemistry of: 89MI19.

Total synthesis of pyrrole pigments: 84MI33.

d. *Porphyrins and Related Systems*. Aminoacyl derivatives of porphyrins and metalloporphyrins: 87MI60.

Binary porphyrins as catalysts: 84MI1.

Hematoporphyrins as photosensitizers: 90WCH149.

Heme catabolism: 87ACR250.

Hemoglobin, stereochemistry of cooperative mechanisms in: 87-ACR309.

Hydroporphyrins: 88AHC(43)73.

Iron-porphyrins, Mössbauer spectra, and electronic structure of: 89-ZSK148.

Iron-porphyrins, synthesis and coordination properties of: 86PAC1493.

Magnetic circular dichroism spectra of π -substituted porphyrins: 88-ACR95.

Metalloporphyrins as initiators of vinyl polymerization: 89YGK1017.

Metalloporphyrins, aspects of organometallic chemistry of: 88-CRV1121.

Metalloporphyrins, monographs: 87MI27; 88MI45.

Microbial synthesis of porphyrins and corrinoids: 87MI50; 89MI6.

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Nomenclature of tetrapyrroles: 88MI60.

Organocobalt coenzyme B₁₂ models: 89CSR225.

Polymer-bound heme derivatives, physiological behavior of: 88-YGK879.

Porphyrin derivatives, synthesis and application of: 88YGK681.

Porphyrin complexes, oxygenation of hydrocarbons with: 89ZC88.

Porphyrin molecular complexes, photonics of triplet states of: 88-UK1087.

Porphyrin-quinone compounds as models of photosynthesis reaction center: 89UK1032.

Porphyrins, annual report: 89AR(B)321.

Protoporphyrin-IX, structural modifications: 87H(26)1947.

Sandwich-compounds of metals with porphyrins: 90MI42.

Spectroscopy, electrochemistry, and applications of porphyrins, monograph: 87MI18.

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e. *Indoles and Hydroindoles*. N-Acylizatin ring expansion: 88MI28.

Alkylindoles, syntheses of: 87KGS1155.

¹³C NMR spectroscopy of indoles: 87MI38.

Cyanohydrin-*O*-diethyl phosphates in indole chemistry: 88YGK1165.

Fischer indole synthesis, mechanism of: 88KGS867.

Heterogeneous catalysis of indolization of arylhydrazones: 88KGS-1443.

Indigo derivatives, synthesis and application of: 88YGK681.

Indole-2-carboxylic acid, chemistry of: 87YGK1171.

Indole-2,3-quinodimethanes and their analogues in synthesis of [*b*]-annelated indoles: 89CRV1681.

2-Methyleneindoline bases, chemistry of: 90MI19.

Nitroindoles: 86MI11.

Oxidation of indoles in the CNS, electrochemical studies of: 90CRV795.

Transition metals in the synthesis and functionalization of indole: 88AG(E)1113.

Tryptophan in asymmetric synthesis: 87MI25.

Tryptophan in peptide synthesis: 90OPP393.

Tryptophan interaction with biologically important molecules: 88-YZ506.

Tryptophan metabolites, biomimetic synthesis of: 86YZ964.

Vinylindoles, Diels-Alder reactions as a route to annelated indoles: 88H(27)1253.

f. *Isoindoles (Including Phthalocyanins)*. 3-Amino-1-imino-1H-isoin-
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Phthalimide, nucleophilic nitrosubstitution in the synthesis of polyim-
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Phthalocyanins, advances in the chemistry of: 86PAC1467.

Phthalocyanins, applications to functional materials: 87YGGK837.

Polymer phthalocyanins, coordination compounds of: 87MI45.

Sandwich compounds of metals with phthalocyanins: 90MI42.

g. *Carbazoles*. *N*-(2,3-epoxypropyl)carbazoles: 90UK738.

Formylcarbazoles, synthesis and application of: 87MI46.

h. *Polycyclic Systems Including Two Heterocycles*. Furo[3,2-*b*]pyr-
roles: 90CCC597.

Fuzed azino[*a*]indoles: 90KFZ(5)9.

Indolizines, mass spectral fragmentation of: 90MI41.

Perhydro azino[*a*]pyrroles: 90AHC(49)193.

Pyrrolizidines, asymmetric synthesis of: 90H(30)1231.

Pyrrolizidines, syntheses of: 88H(27)1465.

Pyrrolizine chemistry: 87S10.

Pyrrolo[2',3' : 4,5]furo[3,2-*b*]indoles: 90CCC597.

3. *One Oxygen Atom*

a. *Furans*. Chlorinated furans, environmental behavior of: 90-
ACR194.

Cycloaddition reactions of furan derivatives: 83MI6.

Electrochemical functionalization of furan halides: 90S369.

Furan derivatives in the synthesis of benzenoid molecules by low-valent-
titanium deoxygenation: 89ACR145.

Furans in the synthesis of carbohydrates: 84MI9.

Furan synthons for side chains of steroids: 90YGGK43.

Furfuryl alcohol in the synthesis of cyclopentenolones: 90YGK119.

Hydrogenation of furans using Ni–Al alloy: 89CRV459.

Macromolecular furan derivatives: 83MI7.

Nitrofurans, chain electron-transfer reactions of: 83MI8.

Nitrofurans, nucleophilic substitution of: 83MI9.

Photochemical arylation of furans: 89G419.

Photooxidation of furans: 87RTC469.

Silicon and germanium derivatives of furan: 87MI4; 90MI39.

Transition metal-catalyzed cycloaddition reactions of alkynes in syntheses of furans: 88CRV1081.

b. *Hydrofurans*. Carbohydrate derivatives in the synthesis of natural tetrahydrofuran compounds: 84MI10.

Chlorotetrahydrofurans 87MI48.

c. *Benzannelated Furans*. Isobenzofurans in the synthesis of natural products and polyaromatic hydrocarbons: 88T2093.

Polychlorinated dibenzofuran derivatives, analysis in environmental objects: 90UK1799.

d. *Terpenoids Including Five-Membered Ring with One Oxygen Atom*. Alanto- and isoalantolactones: 90KPS307.

Furanoditerpenoids from *Teucrium* species, chemistry of: 87H(25)-807.

Natural polycyclic γ -lactones, retrosynthetic analysis and synthesis of: 88CSR111.

e. *Miscellaneous Bi- and Polycyclic Systems*. 2,3-Bis-(methoxycarbonyl)-7-oxanorbornadiene, retro Diels-Alder reaction with dienes under high pressure: 90YGK132.

Optically pure 7-oxabicyclo[2.2.1]hept-5-en-2-yl derivatives as new chirones: 90MI55.

f. *Five-Membered Lactones*. $\Delta^{\beta,\gamma}$ -Butenolides, general review: 87-KGS723.

Hydrofuranones, synthesis from furans and application in organic synthesis: 83MI5.

α -Methylene- γ -lactones, synthesis using reaction of activated vinyl carbanions with aldehydes: 88T4653.

Photochemistry of 2(3*H*)- and 2(5*H*)-furanones: 90H(31)751.

Transition metal-catalyzed reactions of alkynes in the synthesis of furanones and furandiones: 88CRV1081.

4. *One Sulfur Atom*

a. *Thiophenes*. General monograph: 86HC(44,2)1, 86HC(44,3)1.

Alkylthiophenes: 86HC(44,2)119.

Aminothiophenes: 86HC(44,2)631.

Catalytic synthesis of thiophenes: 88MI16; 90MI12.

Desulfurization of thiophenes: 88MI17; 89CRV459; 90MI13.

Electrochemical functionalization of halothiophenes: 90S369.

Electrophilic substitution of thiophenes: 86HC(44,2)1.

Gewald reaction in the synthesis of bioactive thiophene derivatives: 90PHA545.

Formyl and acyl derivatives of thiophene: 86HC(44,3)309.

Halothiophenes: 86HC(44,2)159.

Hydrodesulfurization of thiophenes in petroleum, mechanism of: 88-ACR387.

Hydroxythiophenes and related compounds: 86HC(44,3)1.

Mass spectrometry of thiophenes: 88MI19; 90MI15.

Metallation of thiophenes: 88MI14; 90MI10.

Nitrothiophenes: 86HC(44,2)523.

Photochemical arylation of thiophenes: 89G419.

Thermal reaction of halothiophenes with thiyl radicals: 90UK1338.

Side-chain reactivity of thiophenes: 86HC(44,3)975.

Thiophene oligomers, preparation of: 88H(27)1731.

Thiophenecarboxylic acids and their derivatives: 86HC(44,3)565.

Thiophenethiols, thienyl sulfides, and related compounds: 86HC-(44,3)135.

Thiophenes of the type found in petroleum, syntheses of: 88MI8.

Thiophene derivatives, synthesis from acetylenes or allenes and thiocarbonyl compounds: 87MI34.

S-Thiophenium salts: 89AHC(45)151.

Thiophenium ylides: 87ACR18; 89AHC(45)151.

Transition metal-catalyzed synthesis of thiophenes: 88MI24.

b. *Annelated Thiophenes*. Aromaticity of annelated thiophenes: 88CCC2023.

Benzo[*b*]thiophene-2,3-dione, chemistry of: 88H(27)1489.

Gewald reaction in the synthesis of annelated thiophenes as biologically active substances: 90PHA545.

Mass spectrometry of annelated thiophenes: 88MI19; 90MI15.

Peri-condensed thiophenes: 90H(30)1219.

Thieno-annelated tetracyanoquinodimethanes and dithiadiselenafulvalenes as organic conductors: 89PS(43)187.

c. *Hydrothiophenes*. Catalytic synthesis of tetrahydrothiophene, sulfolane, and sulfolenes: 88MI16; 90MI12.

3-Sulfolenes as precursors of 1,3-dienes: 88YGK893; 89PP257.

C. TWO HETEROATOMS

1. General

Basicity and acidity of azoles: 87AHC(41)187.

2-Chloromethyl-1,3-azoles, reactions with sulfur: 88ZC233.

Diazoazoles: 90AHC(48)66.

Functionally substituted azoles, new approaches to synthesis of: 89KGS723.

Immunotropic activity of azole derivatives: 90KFZ(10)19.

Nitroazoles, general monograph covering N- and N,O-azoles: 86MI11.

Organometallic (Li, Na, Mg) derivatives of azoles: 88KGS147.

Oxidative transformations of azole iminium salts: 87AHC(41)275.

Perhydro bicyclic azoloazines with bridgehead N atom: 90AHC(49)193.

Trifluoromethyl-substituted azoles, synthesis of: 87YGK269.

2. Two Nitrogen Atoms

a. *Pyrazoles*. Nitropyrazoles: 88KGS435.

Pyrazole 1-oxides, 1,2-dioxides, and derivatives: 89H(29)1615.

b. *Hydropyrazoles*. Aryl-substituted 2-pyrazolines, structure, luminescent and other photochemical properties of: 89MI23.

Pyrazolines, synthesis from aliphatic diazocompounds: 85MI4.

5-Pyrazolone couplers for color photography: 87YGK151.

c. *Annelated Pyrazoles*. Pyrazoles, condensed with 5- and 6-membered heteroaromatic rings: 90AHC(48)223.

Pyrazolopyrimidines: 87AHC(41)319.

d. *Imidazoles*. Imidazole derivatives as inhibitors of tromboxane- A_2 synthase and antagonists of tromboxane- A_2 receptor: 87YGK1.

Imidazole derivatives as models of enzymes: 87ACR146.

Imidazoles activated by sulfur functional groups, reactivity of: 87YGK624.

β -Imidazolylenones, synthesis and reactions of: 87YGK863.

Nitroimidazoles as chemoterapeutic agents: 83MI4.

Organolithium derivatives of imidazole: 88MI30.

Phosphorylated imidazoles: 90KGS723.

1-Substituted imidazoles as antifungal agents: 83MI4.

Synthesis of imidazoles from hydrogen cyanide derivatives: 87-AHC(41)1.

e. *Hydroimidazoles*. Histidine in peptide synthesis and its imidazole function: 89OPP393.

Hydantoins, chemical properties of: 88AKZ548.

Imidazole-2-thiones, structure and properties of: 88KGS1587.

Imidazolidinyl protecting groups for α -aminoaldehydes: 89CRV149.

Imidazoline nitroxyl radicals, monograph: 88MI3.

f. *Annelated Imidazoles*. Benzimidazoles and congeneric tricyclic systems, general monograph: 80HC(40,2)1; 81HC(40,1)1.

Benzimidazole anthelmintics: 83MI4.

Benzimidazoles: 81HC(40,1)1.

Benzimidazole-N-oxides: 81HC(40,1)287.

Condensed benzimidazoles of the type 5-6-5: 81HC(40,1)391.

Condensed benzimidazoles of the type 6-6-5: 81HC(40,1)483.

Condensed benzimidazoles of the type 6-5-5: 80HC(40,2)1.

Condensed benzimidazoles of the type 6-5-6; 90HC(40,2)257.

Condensed benzimidazoles of the type 6-5-7 and higher homologues: 80HC(40,2)463.

Condensed benzimidazoles with bridges between 1-N and 7-C: 80HC(40,2)505.

Condensed imidazoles of the type 5-5: 86HC(46)1.

Dihydrobenzimidazoles, benzimidazolones, benzimidazolethiones, and related compounds: 81HC(40,1)331.

Imidazoquinazolines, synthesis, reactivity, biological activity of: 90-AKZ245.

Levamisole (imidazo[2,1-*b*]thiazole drug), synthesis and chemical properties: 89KFZ206, 89KFZ801.

Phosphorylated annelated imidazoles: 90KGS723.

Practical applications of benzimidazoles: 80HC(40,2)531.

Purines, see Section VI,C,1,e.

Thiazolobenzimidazoles: 88H(27)1975.

3. One Nitrogen and One Oxygen Atom

a. *1,2-Heterocycles*. Cycloaddition reactions of nitrile oxides, nitrones and nitronates, synthetic use of: 88MI57.

Cycloaddition of nitrile oxides and nitrones to alkenes, stereocontrol in: 89G253.

Cycloaddition of nitrones: 86MI17; 88OR(36)1.

Isoxazole derivatives, synthesis, chemical transformations, use in total synthesis of natural products: 87S857; 89KGS435.

Isoxazolines, photochemistry of: 88MI29.

Isoxazolines, stereoselectivity of formation by cycloaddition to chiral enitols and pentenolides: 88MI32.

Isoxazolones, electron transfer ring opening reactions of: 89YGK629.

Oximes of 3-acylisoxazoles, rearrangement into furazans: 90KGS1443.

b. *1,3-Heterocycles*. 4-Alkoxy carbonyloxazoles as α -amino- β -hydroxy acid synthons: 88H(27)1035.

4-Alkylideneoxazolidine-2,5-diones in peptide synthesis: 89YGK782.

Chiral oxazolines in asymmetric synthesis: 84MI12.

1,2-Dideoxyhexopyrano[2,1-*d*]oxazolines and respective oxazolinium salts, syntheses and reactions of: 89MI33.

Oxazolidine-2,5-diones(N-carboxy α -amino acid anhydrides), general monograph: 87MI58.

Oxazolidine-2-one protecting groups in stereoselective aldol reactions: 87AG24.

2-Oxazolones as synthons: 87H(26)1077; 88CSR91, 88YZ593.

Silicon and tin derivatives of oxazoles as synthons: 88G211.

4. One Nitrogen and One Sulfur Atom

a. *1,2-Heterocycles*. Benzo[*d*]isothiazole-1,1-dioxide derivatives as activators of polycondensation of dicarboxylic acids with diamines: 90YGK144.

b. *1,3-Heterocycles*. 2-(2-Aminothiazol-4-yl)-2-hydroximinodiacetic acid derivatives, synthesis of: 88MI64.

Chiral thiazolidines: 90YGK986.

Condensed 4-thiazolidinones: 90AHC(49)3.

Levamisol (imidazo[2,3-*b*]thiazole drug) and its analogues, synthesis and reactions of: 89KFZ206, 89KFZ801.

Silicon and tin derivatives of thiazole as synthons: 88G211.

Thiazole and benzothiazole derivatives activated by sulfur functional groups, reactivity of: 87YGK624.

Thiazoles as intermediates for carbohydrates and biologically active derivatives: 89PS(43)25.

Thiazolidine-2,5-diones: 87MI58.

Thiazolobenzimidazoles: 88H(27)1975.

Thiazolo[3,2-*a*]pyrimidines: 89MI10.

5. *Two Oxygen Atoms*

1,3-Dioxolanes, heterolytic ring cleavage with Grignard reagents: 90UK401.

1,3-Dioxolanium ions: 87CSR75.

6. *One Oxygen and One Sulfur Atom*

1,3-Oxathiolane derivatives in the study of muscarinic agonists and competitive antagonists: 89FES897.

γ -Sultones: 87T1027.

7. *Two Sulfur Atoms*

a. *1,2-Heterocycles.* 1,2-Dithiol-3-thiones, formation in 1,3-anionic cycloaddition reactions of α,β -unsaturated thiolates: 87UK267.

Poly-(1,2-dithiolo[4,3-*c*]-1,2-dithiol)-2,6-ylidene as a potential conducting material: 89PS(43)165.

1,2-Tetrathiafulvalenes, synthesis, chemical and physical properties of: 87MI61.

b. *1,3-Heterocycles.* Benzo-1,3-dithiol-2-yl as a protecting group in the synthesis of nucleic acid derivatives: 87YGK930.

1,3-Dithiolate conducting complexes, molecular structures of: 90UK-1179.

1,3-Dithiol-2-imines, formation in 1,3-anionic cycloaddition reactions of α,β -unsaturated thiolates: 87UK267.

1,3-Dithiol-2-thione, formation from tetrathioxalates: 86MI28.

1,3-Tetrathiafulvalenes, synthesis, properties of: 87MI61.

Tetrachalcogenofulvalenes and their charge-transfer complexes, conducting properties and nature of heteroatoms: 87YGK502.

D. THREE HETEROATOMS

1. *General*

Basicity and acidity of azoles: 87AHC(41)187.

2. *Three Nitrogen Atoms*

a. *Triazoles*. Nitrotriazoles: 86MI11.

4-Amino-1,2,3-triazoles: 86AHC(40)129.

Organolithium derivatives of 1,2,3-triazoles: 88MI30.

1,2,3-Triazole-1-oxides, synthesis and properties of: 89KGS147.

1,2,4-Triazole derivatives with antifungal activity: 83MI4.

b. *Hydrotriazoles*. 1,2,4-Triazolines: 89AHC(46)170.

c. *Annelated Triazoles*. 8-Azapurines (1,2,3-triazolo[4,5-*d*]-pyrimidines: 86AHC(39)117.

Benzotriazoles, synthesis, properties, applications: 87MI44.

Synthesis of fused 1,2,4-triazolo[3,4-*z*]heterocycles: 90AHC(49)277.

3. *Two Nitrogen Atoms and One Oxygen Atom*

Furazans, synthesis of using rearrangement of 3-acylisoxazole oximes: 90KGS1443.

Fused furoxans: 99MI59.

Nitrooxadiazoles: 86MI11.

1,2,4-Oxadiazoles, synthesis and reactivity of: 88MI157, 88Y GK256.

4. *Two Nitrogen Atoms and One Sulfur Atom*

Condensed systems including the 1,2,5-thiadiazole ring: 89Y GK1108;

EPR spectra of 1,2,5-thiadiazole radicals: 90MI64.

X-Ray fluorescence spectra of 2,1,3-thiadiazole derivatives: 88MI21; 90MI17.

Δ^4 -1,2,4-Thiadiazolines, synthesis and reactivity of: 88Y GK256.

5. *One Nitrogen Atom and Two Sulfur Atoms*

EPR spectra of 1,2,3-, 1,2,5-, and 1,3,2-dithiazole free radicals: 90MI64.

E. FOUR HETEROATOMS

1. *Four Nitrogen Atoms*

Basicity and acidity of tetrazoles: 87AHC(41)187;

Nitrotetrazoles: 86MI11;

Synthesis of tetrazoles from aliphatic diazo compounds: 85MI5;

Tetrazolium salts: 90KGS1587.

2. *Two Nitrogen Atoms and Two Sulfur Atoms*

EPR spectra of 1,3,2,4-dithiadiazole free radicals: 90MI64.

VI. Six-Membered Rings

A. GENERAL

3-Aminothioacrylamides in synthesis of mono- and diazines: 87ZC8.

Antiaromatic azacycl[3.3.3]azines, chemistry of: 87H(26)2757.

Azines as pesticides, syntheses of: 89MI1.

Azinium halochromates and bichromates as oxidants for organic synthesis: 88OPP533.

Azinium salts, oxidative transformations of: 87AHC(41)275.

Azino[a]indoles, synthetic routes to: 90KFZ(5)9.

Azinylisocyanates and isothiocyanates, synthesis and reactions of: 87S525.

Azinyl-ylidene tautomerism of azinylmethanes and general problems of azine tautomerism: 90UK456.

Conformational analysis of 6-membered saturated S-heterocycles: 89ACR357.

Cycloaddition reactions of 6-membered heteroaromatics: 89CRV827.

Hydrogenation of azines using Ni-Al alloy as a reducing agent: 89CRV459.

Nucleophilic substitution of hydrogen in azines: 88T1.

Partially hydrogenated azines as membranoprotectors and Ca ions antagonists: 87MI7.

Photochemistry of azines: 90UK279.

Regioselective nucleophilic, electrophilic, and radical substitution in pyridines, di-, tri-, and tetrazines: 88AHC(44)199.

B. ONE HETEROATOM

1. *One Nitrogen Atom*

a. *Pyridines.*

i. *Structure.* Pyridine-metal complexes, general monograph in three parts: 85CHE(14,6)1.

Structural studies of pyridine derivatives: 87MI11.

ii. *Reactivity*. Amination of pyridines: 8/KGS1011; 88AHC(44)1; 90AHC(49)117.

4-(Dialkylamino)pyridines as catalysts in organic synthesis: 86-CLY1071.

Dipyridyl ethers, sulfides, and selenides: 87JHC533.

Free-radical substitution of pyridines: 79ZVK134; 89H(28)489; 90-JHC79.

Fluorination of pyridine derivatives: 88OR(35)513; 89YGK619; 90-CLY952.

N-Hydroxy-2-thiopyridone, radical reactions of: 89PS(43)349; 90-YGK641.

Metallation and metal-assisted bond formation in pyridines: 90-H(30)1155.

Pyridines, reactions with nucleophilic reagents: 90UK888.

Pyridylsilanes in synthesis of heterocyclic derivatives: 87KGS5.

2-Pyridyl-substituted compounds, synthetic reactions with: 88OPP145.

Pyridylsulfides, reactions of: 87MI63.

Pyridyl compounds activated by sulfur functional groups, reactivity of: 86MI29; 87YGK624.

Thermal and photochemical behavior of cyclomers of ethylenebis- and trimethylenebis(pyridyl)diradicals: 90H(30)1307.

iii. *Synthesis*. Coordination in synthesis of pyridine derivatives: 87WCH741.

Diazotization-fluorodediazoniatio of aminopyridines and synthesis of fluoropyridines: 89YGK619.

Dipyridyl ethers, sulfides, and selenides: 87JHC533.

Halopyridines, synthesis of: 87MI2.

Optically active disubstituted pyridines: 89G71.

2(1H)-Pyridinethiones, synthesis and chemistry of: 88SR(8)105.

2-Pyridone derivatives, synthesis using ketene dithioacetals: 89-YGK413.

Pyridyl sulfides, syntheses of: 87MI63.

Transition metal-catalyzed syntheses of pyridines: 88CRV1081; 88MI24, 90AHC(48)177.

Vicinally substituted nitropyridine derivatives: 87H(26)2727.

b. *Pyridinium Compounds, Ylides, Pyridine N-Oxides*. N-Alkylpyridinium salts, addition reactions with stabilized C-nucleophiles and their applications to alkaloid synthesis: 88H(27)789.

Luminescent spectroscopy and photochemistry of pyridinium salts: 87H(26)2963.

Pyridinium cations, role in mechanisms of nucleophilic aliphatic substitution: 90CSR83.

Pyridinium ylides: 79ZVK496.

N-Substituted pyridinium salts: 89H(29)557.

Vicinally substituted nitropyridine N-oxides, synthesis of: 87H(26)2727.

c. *Applications of Pyridines.* Halopyridines, application of: 87MI2.

Filmforming electropolymerization of pyridines: 87CLY673.

Pyridines with phototropic properties: 88MI34.

d. *Bipyridines.* 6,6'-Diamino-2,2'-bipyridines as metal chelating agents: 87YGK462.

Electroanalytic chemistry of 2,2'-bipyridine complexes: 87ZAK787.

e. *Hydropyridines.* Dihydropyridine derivatives, reduction of with chiral reagents: 83MI1.

1,4-Dihydropyridines, reactions of: 88H(27)291.

1,4-Dihydropyridines, synthesis of by cyclocondensation reactions: 88H(27)269.

1,4-Dihydropyridines, synthesis of: 90KFZ(6)14.

Piperidine-4-carboxaldehydes, ketones, acids, and their derivatives, syntheses of: 89AKZ99.

Tetrahydropyridinium salts, stereochemistry of nucleophilic addition to: 84MI28.

f. *Biologically Active Pyridines and Hydropyridines.* 1,4-Dihydropyridines, pharmacological activity of: 90KFZ(6)14.

Pyridine derivatives as inhibitors of thromboxane- A_2 -synthase and agonists of thromboxane- A_2 receptor: 87YGK1.

Pyridine derivatives as pesticides: 89KGS579, 89MI1; 90MI1.

Pyridyl sulfides, biological activity of: 87MI63.

g. *Pyridines Annelated with Carbocycles.* Annelated pyridines, synthesis using catalytic dehydrocyclization of substituted pyridines: 88-KGS1011.

Decahydroquinoline and decahydroquinolone, stereochemistry of: 87KGS579.

1,2-Dihydroisoquinolines and related compounds: 86AHC(40)105.

8-Mercaptoquinoline and its derivatives, coordination, physical and analytical chemistry of: 87MI42.

8-Mercaptoquinoline chelates, molecular and electron structures of: 87MI41.

8-Mercapto-2-substituted quinolines, structure and properties of chelates: 90MI37.

1,10-Phenanthroline complexes, electroanalytic chemistry of: 87-ZAK787.

Quinolyl compounds activated by sulfur functional groups, reactions of: 86MI29; 87YGK624.

h. Pyridines Annellated with Heterocycles. Azaadamantanes with bridgehead N-atoms: 89UK1815.

Azacycl[3,3,3]azines: 87H(26)2757.

Indolizines, mass spectral fragmentation of: 90MI41.

Isoquinuclidine (2-azabicyclo[2,2,2]octane), catalytic synthesis of: 88MI25.

Perhydro azolopyridines with bridgehead N-atoms: 90AHC(49)193.

Pyranopyridines, chemistry of: 87AKZ104.

Pyrido[2,3-*d*]pyrimidines: 87UK2001.

Praziquantel, hexahydropyrazino[2,1-*a*]isoquinoline derivative with potent anthelmintic activity: 90KFZ(9)60.

Quinuclidine derivatives, synthetic chemistry, and pharmacology of: 83MI4.

2. One Oxygen Atom

a. Pyrylium Compounds. Pyrylium cations in nucleophilic substitution: 90CSR83.

b. Pyrans and Hydropyrans. 3-Amino-2,3,6-trideoxyhexoses, diastereoselective syntheses of: 89H(28)1229.

3,6- and 5,6-Dihydro-2H-pyrans, reactivity of: 88KGS291.

Glycosylthio-, seleno-, and tellurophosphates: 89H(28)1249.

Halotetrahydropyrans, reactivity of: 89AKZ571.

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1,3-Dioxin-4-ones, synthesis of: 88Y GK596, 88YZ805.

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- c. *Reactivity.* Chlorocyclophosphazenes, reactions with bifunctional reagents: 90H(31)2231.
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B,N-Heterocycles in the synthesis of boron nitride: 90CRV73.
B,P-Heterocycles: 90AG(E)449.
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C. SILICON, GERMANIUM, TIN, AND LEAD HETEROCYCLES

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2. *Structure and Stereochemistry*

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3. *Reactivity*

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4. *Synthesis*

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3. *Reactivity*

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4. *Synthesis*

1,3-Anionic cycloaddition reactions of α,β -unsaturated selenolates and tellurolates in the synthesis of selenophene, tellurophene, and 1,3,4-selenadiazine derivatives: 87UK267.

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E. OTHER UNUSUAL HETEROCYCLES

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Cage compounds with both main group metal and nonmetal atoms: 89PS(41)195.

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Unsaturated metallacycles with main group metals: 87AG1.

Zirconium and hafnium heterocycles: 86MI19.

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Uracils: Versatile Starting Materials in Heterocyclic Synthesis

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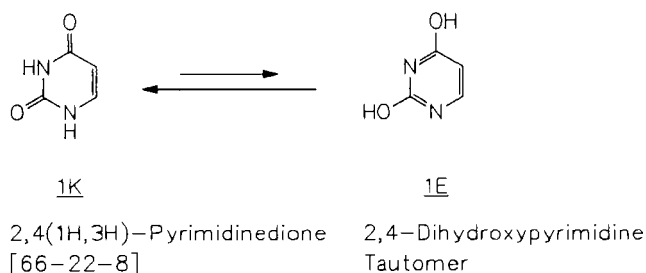
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I. Introduction

2,4(1*H*,3*H*)-Pyrimidinedione normally called by the trivial name Uracil has been known since 1900 when it was first isolated by hydrolysis from materials containing ribonucleic acids, such as yeast (1900ZPC161), wheat germs [02ZPC(36)85], and herring sperm [02ZPC(37)246]. Thymine was found much earlier from bovine thymus (1893CB2753; 1894CB2215). In 1901, the constitution of uracil was established by Emil Fisher (01CB-3751); however, 6-methyluracil was made as early as 1885 (1885LA8) (Scheme 1).



SCHEME 1

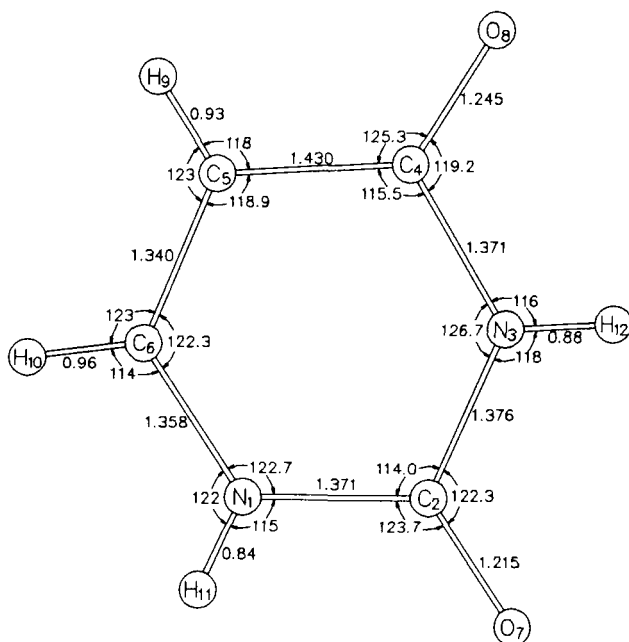
No exhaustive and detailed review on uracils, their syntheses, structure, or their utility in heterocyclic chemistry exists. However, some aspects of uracil chemistry have been discussed within several review series: [62CHE(16)256; 69AHC149; 70CHE(16,S1)193; 76MI1; 80-82MI1; 80MI3; 82MI2; 85CHE(16S2)248,262; 87CHE(47)1].

II. Structure and Physical Properties

In solid state, uracil exists as the dioxo tautomer **1K**, which has been shown with the aid of refined X-ray analyses from which the position of hydrogen atoms were directly determined (54AX313; 67AX1102).

Uracil crystallizes in the space group *P*2₁/*a*. The following list shows some parameters for the monoclinic cell (Scheme 2):

$$\begin{aligned}
 a &= 11.938 \pm 0.001 \text{ \AA} \\
 b &= 12.376 \pm 0.009 \\
 c &= 3.6552 \pm 0.003 \\
 \beta &= 120^\circ 54' + 0.4' \\
 \lambda (\text{Mo } K\alpha) &= 0.71069 \text{ \AA}
 \end{aligned}$$

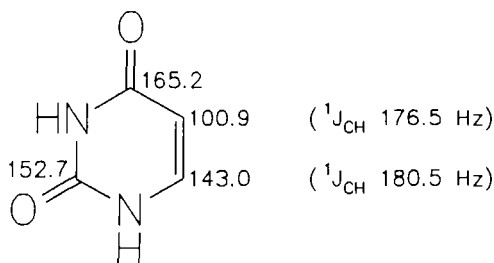


SCHEME 2. Dimensions of a uracil molecule (67AX1102).

This dioxo form is further supported by other spectroscopic data. for instance, UV- (61JCS504) and Raman spectroscopy [67SA(A)2551] indicate that the same dioxo tautomer predominates in solution.

In ^1H -NMR spectroscopy (solvent: D_2O), 5-*H* and 6-*H* form a quadruplet centered at δ 5.71 and 7.60, respectively, with a coupling constant of $J = 8$ Hz (63MI1).

As the chemical shifts of C-5 and C-6, ^{13}C -NMR spectroscopy reveal the 5,6-double bond is highly polarized as expected for a heterocyclic enamino carbonyl compound (84MI2; 85AHC299) (Scheme 3).

SCHEME 3. ^{13}C -NMR data and coupling constants (84MI2).

The ^{15}N -NMR data also support the dioxo structure, although all spectra are complicated by extensive, long-range ^{15}N —H coupling and the low solubility of the material in most solvents (65JA5439).

The mass spectrum (MS) 70 eV (65JA4569) of uracil shows a molecular ion at m/z 112, which expels HNCN (43 mass units) and produces a peak at m/z 69 ($\text{C}_3\text{H}_3\text{NO}^+$) and a metastable peak at m/z 42.5 ($112 \rightarrow 69$). The additional fragmentation processes have been studied in detail. Protonation/deprotonation sites have been discussed [see 76AHC(S1)71].

III. Naturally Occurring Uracils; Uracils as Active Principles

As shown in Sec. I, uracils have represented, for more than 90 years, a class of compounds that continually attract organic chemists, biochemists, medicinal chemists, and photobiologists. Uracils were first detected as constituents of ribonucleic acids, from which they were prepared by hydrolysis. Nucleosides derived from uracil are called uridine, pseudouridine, and uridine phosphate, respectively. Recently, uracil moieties were detected in the antibiotic Tunicamycin (85JA7761).

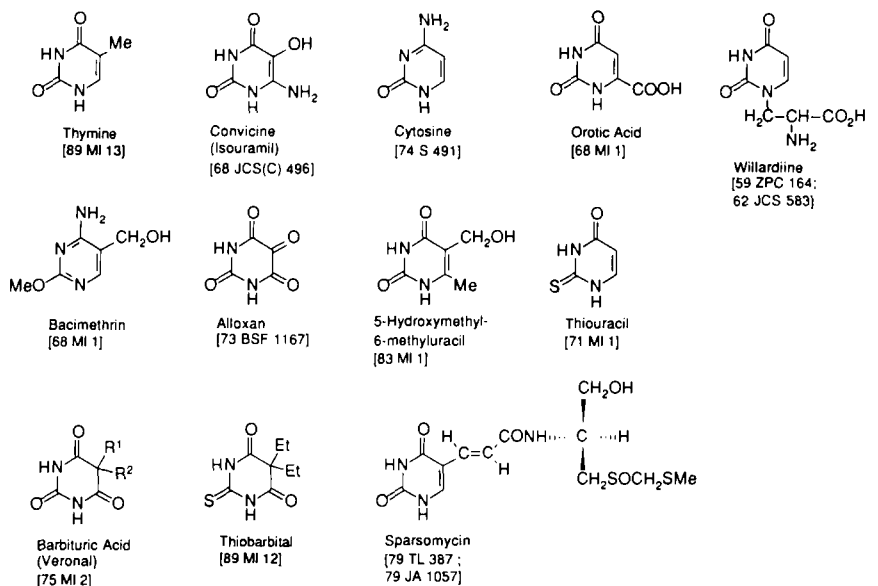
The biosynthesis of uracil proceeds via decarboxylation of orotidin-5'-phosphate, which is formed from carbamoyl phosphate and aspartate via orotate after nucleosidation with 5-phosphoribosyl-1-diphosphate. Uracil can also be generated from cytosine by oxidative deamination using sodium hydrogensulfite.

In Scheme 4, some naturally occurring and synthetic uracils are shown (84MI1), most of them possessing biological activity (Scheme 4).

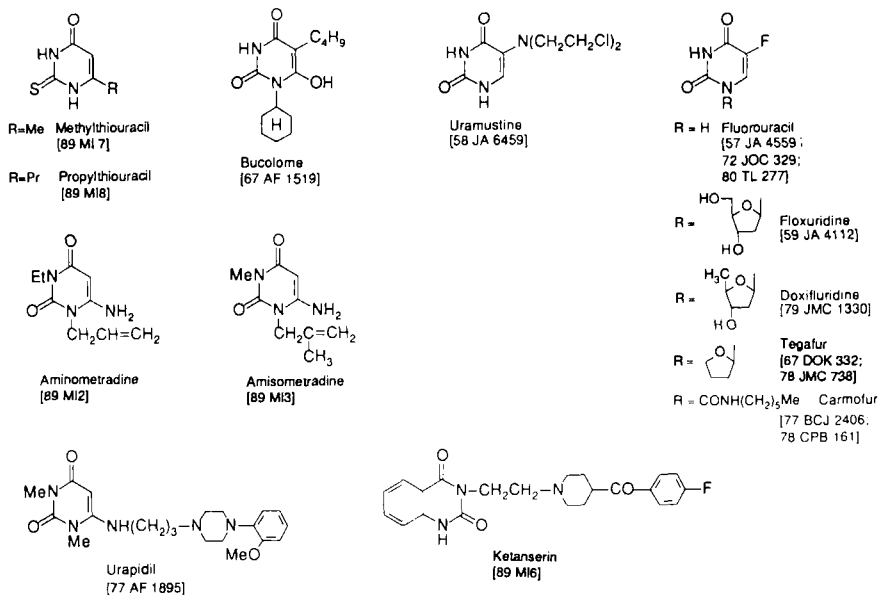
Several uracil derivatives have been developed as drugs. Thus, methylthiouracil and propylthiouracil are thyroid inhibitors; Bucolome is an anti-inflammatory; and Uramustine (Uracil Mustard), Fluorouracil, and its masked compounds are anticancer agents. Aminometradine and Amisometradine are used clinically as diuretics, and Urapidil and Ketanserin are used as antihypertensives (Scheme 5).

Many mono- and bicyclic uracils are used to protect plants, mostly as herbicides (Scheme 6).

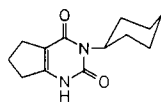
Uracil nucleosides, the uridines and their derivatives, play a decisive role as biologically and pharmacologically active principles. For example, idoxuridine (63JMC428), trifluridine (64JMC1), and edoxuridine (69JMC533; 78JA8106) show antiviral activity as an antimetabolite of thymidine; Cytarabine is used for the clinical treatment of leukemia (65JOC835; 75MI2); and the recently developed Azidothymidine (AZT) (Zidovudine) (64JOC2076; 78MI1; 85MI1, 85PNA7096; 86MI1; 90MI1) and CNT (88JOC4780, 88T625, 88TL941) have been applied successfully



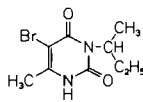
SCHEME 4



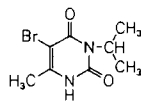
SCHEME 5



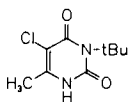
Lenacil, Venzar®



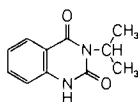
Bromacil, Hyvar X®



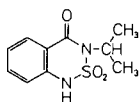
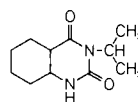
Isocil, Hyvar®



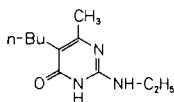
Terbacil, Sinbar®



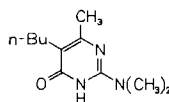
BASF developments



Bentazone, Basagran®



Ethirimol, Milstem®



Dimethirimol, Milcarb®

SCHEME 6. Pyrimidine herbicides.

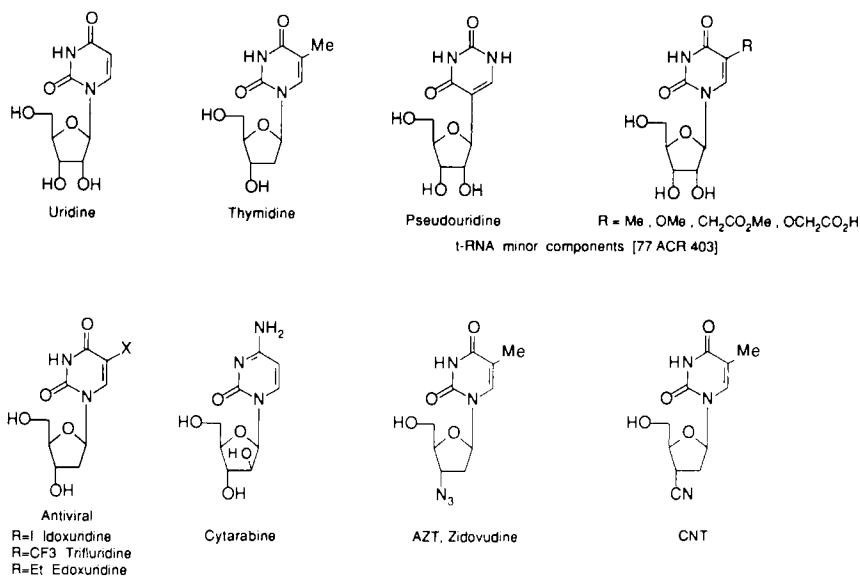
as reverse transcriptase inhibitors (87JMC862, 87MI) in AIDS treatment (Scheme 7).

Naturally occurring heterocondensed uracil derivatives are shown in Scheme 8. Methylxanthines, e.g., caffeine (89MI4), theophylline (89MI11), and theobromine (89MI10) show various pharmacological activities. Riboflavin (Vitamin B₂) acts as a coenzyme in bio-redox reactions (89MI9). Uric acid is a metabolite of purine nucleosides (89MI15). Toxoflavin (62JA1714) and fervenuline (61JOC5256) are antibiotics (Scheme 8).

IV. Uracil Syntheses

The classical and primary synthetic route to uracil from formalacetic acid (made *in situ* from malic acid) and urea in sulfuric acid is still important (26JA2379).

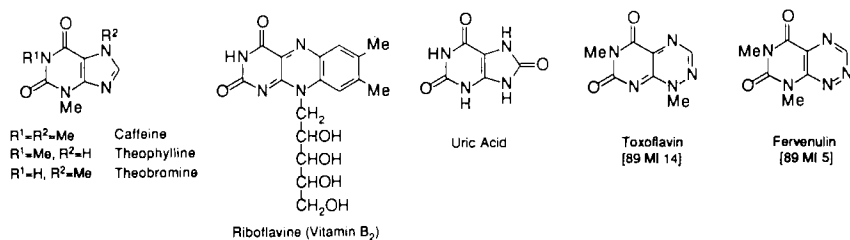
Some alternative syntheses use malic acid, urea, and PPA (61SCI1923) or maleic/fumaric acid, urea, and polyphosphonic acid (PPA) (71S154). The reaction of formylacetate with thiourea is convenient for the synthesis of 2-thiouracil (08JA547; 10JA19). Another main synthesis involves the



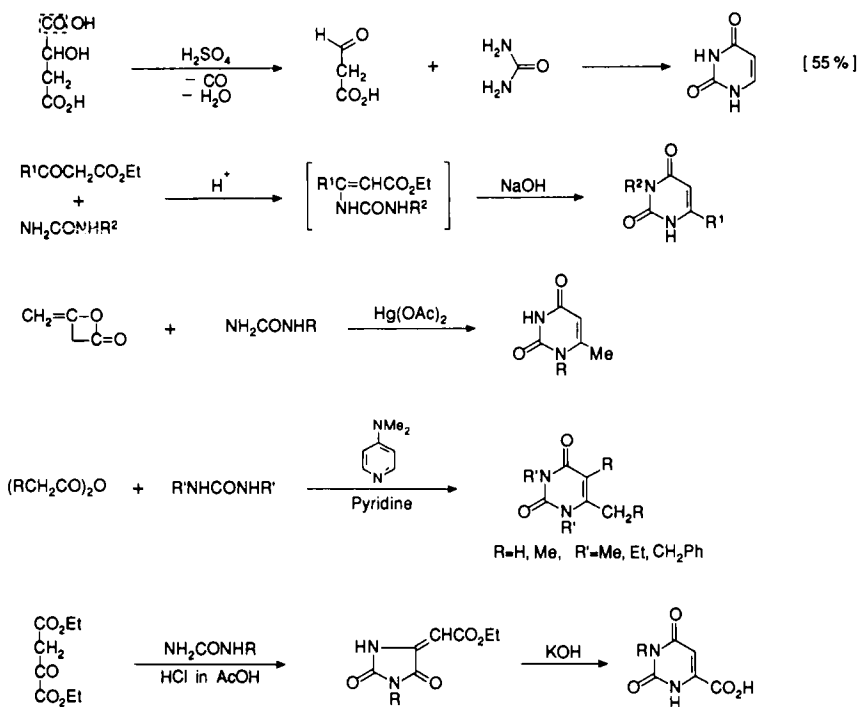
SCHEME 7

reaction of ureas with β -keto esters (48OS422; 58CPB476; 73CPB1894; 74CPB189), diketene (64DOKN1358; 65IZV201; 72JMC471), or acid anhydrides (82S1071). Orotic acids are synthesized from oxaloacetate and ureas in the presence of hydrogen chloride via ring transformation of hydantoin into the uracil ring system (47JA674; 57JCS2367) (Scheme 9).

Treatment of the easily obtainable 2-thiouracil with chloracetic acid followed by acid hydrolysis (08JA547; 52MI1) or by oxidation with dimethylsulfoxide (DMSO) in conc. sulfuric acid (74S491) are alternative pathways. 1,3-Dimethyluracil is transformed with urea in ethanolic sodium ethoxide into uracil (77JHC537; 78JOC1193).

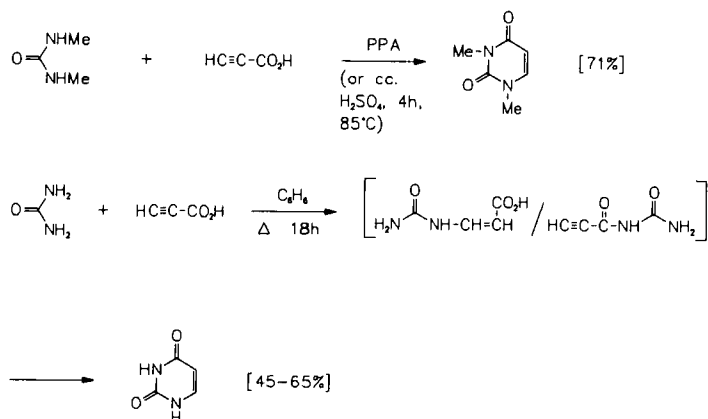


SCHEME 8



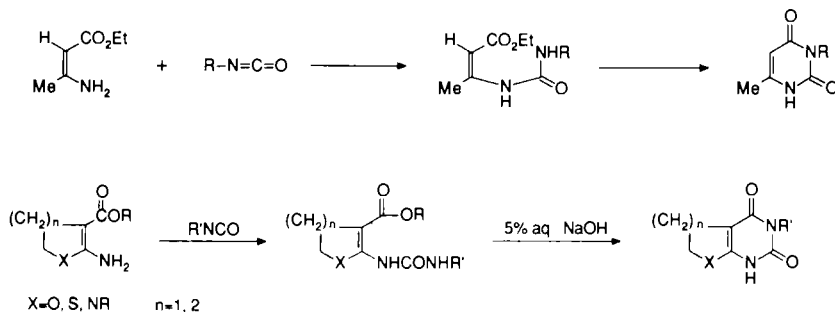
SCHEME 9

Some more recent uracil syntheses start with propiolic acid and urea in PPA (or conc. sulfuric acid and benzene as solvent (76TL2321; 77JOC2185; 89JOC4867) (Scheme 10).



SCHEME 10

A broad choice of heterocondensed uracils are easily and generally accessible from heterocyclic β -enamino esters and isocyanates (01LA200; 03LA341). The mixed urea intermediate is smoothly cyclized with 5% aq. NaOH; the whole procedure can be carried out in a one-step reaction, when pyridine serves as solvent and base catalyst for the ring closure (68CB3377; 85AHC299) (Scheme 11).



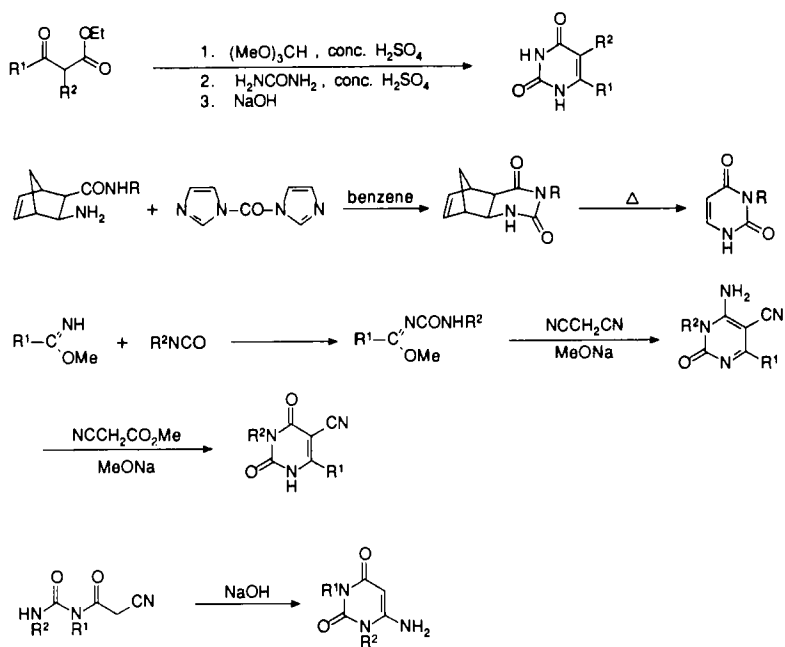
SCHEME 11

The condensation of urea with protected β -ketoesters gives 6- or 5,6-(di)substituted uracils (88MI1). By means of retro Diels–Alder splitting, norbornene condensed tricyclic dihydrouracils, accessible from aminonorbornene carbocyclic acid and 1,1'-carbonyldiimidazole, afford, upon heating, uracils (88MI2) in good yield. Substituted uracils are obtained from imido esters, isocyanates, and malononitrile (88S122). Similarly N' -substituted N -cyanoacetyl ureas cyclize in an alkaline medium (51JOC1879; 89ZC29) (Scheme 12).

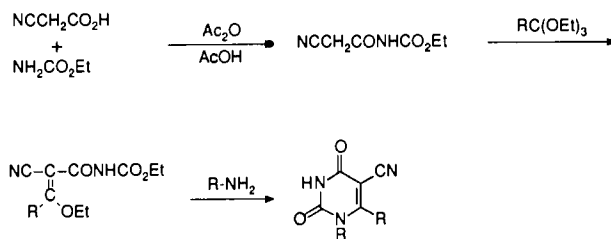
An approach to 5-cyanouracils is accomplished by condensation of O -ethyl- N -cyanoacetylcarbamate with ortho esters and subsequent cyclization using ammonia or primary amines (55JCS1834; 56JCS1877; 56JCS4118). This method is applicable to various routes [53JA671; 55JA5867; 56JA5294, 56JCS3847; 61JCS3254; 68JCS(C)1519; 71JCS(C)2507; 72CPB1380, 72HCA1039; 85JOC4642] (Scheme 13).

Maleic diamide has been cyclized by strong oxidants, lead tetraacetate, or phenyliododiacetate to substituted uracils in high yield (27RTC268; 90AJC451) (Scheme 14).

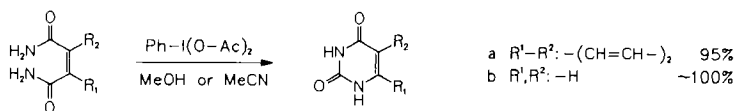
Heterocondensed uracils are easily accessible from acyllactones, -lactams, -thiolactones (72S151), and heterocyclic β -enamino esters, especially (85AHC299). The latter gives a broad range of novel types of condensed systems. With the aid of the hexamethyldisilazane trimethylchlorosilane (HMDS/TMSCl) technique or the use of NaH and halo-sugars, respectively, simple approaches have been developed to obtain unusual nucleosides [92JPR(ip)] (Scheme 15).



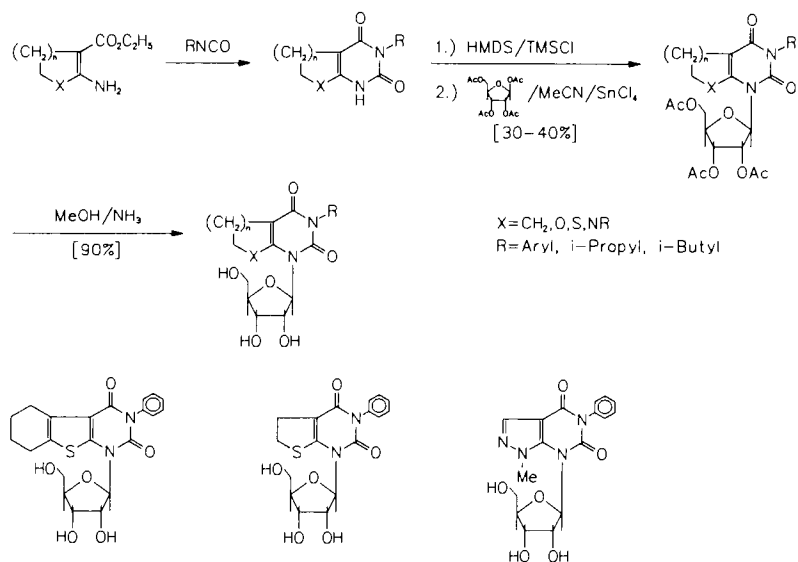
SCHEME 12



SCHEME 13

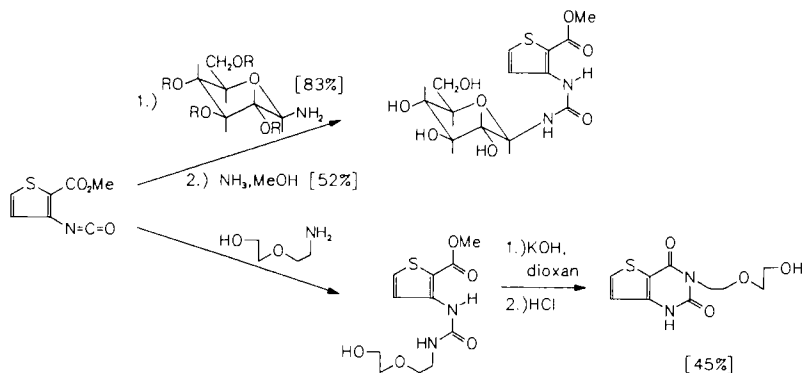


SCHEME 14

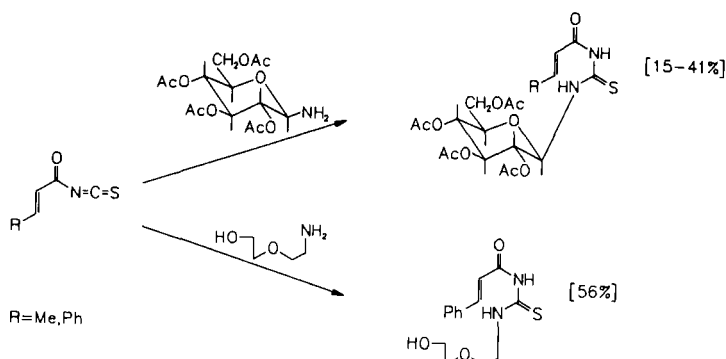


SCHEME 15

Synthesis of another type of acyclo nucleosides start with thiophene-3-isocyanate and aminosugars to afford, with KOH/dioxan, thieno[3,2-*d*]pyrimidine nucleosides, while acryloylisocyanates react readily with aminoglucose or open chain analogues, resulting in open-chain (acyclo)uracil and an acyclo-acyclo species [92S(ip)] (Scheme 16, Scheme 17).



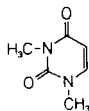
SCHEME 16



SCHEME 17

V. 1,3-Dimethyluracil as a Model Compound

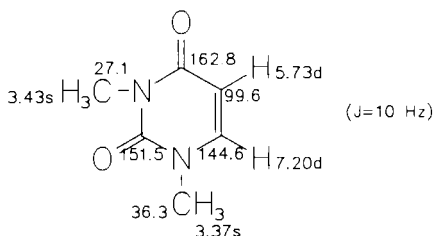
Because of its strongly polar groups, uracil itself shows only a weak solubility in the normal solvents (0.358 parts of uracil in 100 parts of water at 25°C; almost insoluble in alcohol or ether; $pK_a = 9.45$). It is soluble in hot water and in aqueous alkali or ammonia, forming ionized species (cf. Scheme 1). Thus, uracil is, with few exceptions, not suited for chemical reactions in organic solvents. For this reason, 1,3-dimethyluracil [1,3-dimethyl-2,4(1*H*,3*H*)pyrimidinedione] has been chosen by many research groups as a versatile model compound (Scheme 18).



SCHEME 18

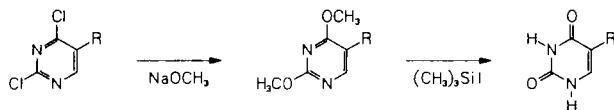
The first synthesis of this compound was reported in 1908, starting from uracil and methyl iodide in alcoholic KOH (08JBC49). Later syntheses proceeded from uracil and diazomethane in ether (30JA1536), or dimethyl sulfate in aq. NaOH (59BBA406). Thermal treatment of 2,4-dimethoxy-pyridine also gives 1,3-dimethyluracil by rearrangement (30CB1974, 30JA2001, 30JA4511). For alkylation of uracils using phase transfer catalysis, see the review by Bram *et al.* (85S543) (Scheme 19).

Scheme 19 shows ^1H - and ^{13}C -NMR data (63M11; 78CJC725) of 1,3-dimethyluracil. Once again the 5,6-double bond shows significant polarization (^{13}C -NMR $\Delta \delta_{5,6} = 45$ ppm).



SCHEME 19

2,4-Dimethoxypyrimidines can be considered model alternatives to 1,3-dimethyluracil. After chemical treatment of the pyrimidine nucleus, both protecting methoxy groups are easily removed with the aid of trimethylchlorosilane to give substituted uracils (82JHC463; 90S243) (Scheme 20).



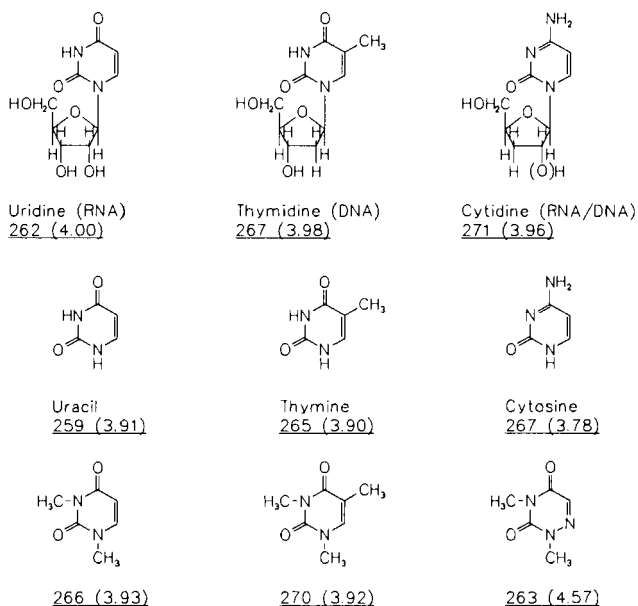
SCHEME 20

VI. Photoreactions of Uracils and Their Derivatives

The photochemical behavior of uracil and its derivatives (e.g., thymine) has been intensively investigated largely because they occur as building units of nucleic acids. Any photochemical changes of these heterocyclic nuclei upon UV irradiation are of special interest for modern photobiology [68MI2; 69AG581; 75HOU(5b)1530; 76M11].

Some related structures shown in Scheme 21 have similar UV absorptions and molar absorptivities (82MI2).

As the classical photo experiment of Beukers and Berends has shown, thymine dimerizes along its 5,6-double bond upon short wavelength irradiation ($\lambda = 253.7$ nm) in an ice matrix to form a *syn*-head-head dimer (60BBA181, 60BBA550). This reaction is a special case of the photodimerization of α,β -unsaturated ketones and carbocyclic acids [75HOU(5a)347]. However, in aqueous solution, all four theoretically possible cycloadducts have been obtained [60N(L)844; 66TL4471; 69M11; 72JA255]. Photolyase of DNA catalyzes the monomerization of the photodimer (89JA9264) (Scheme 22).



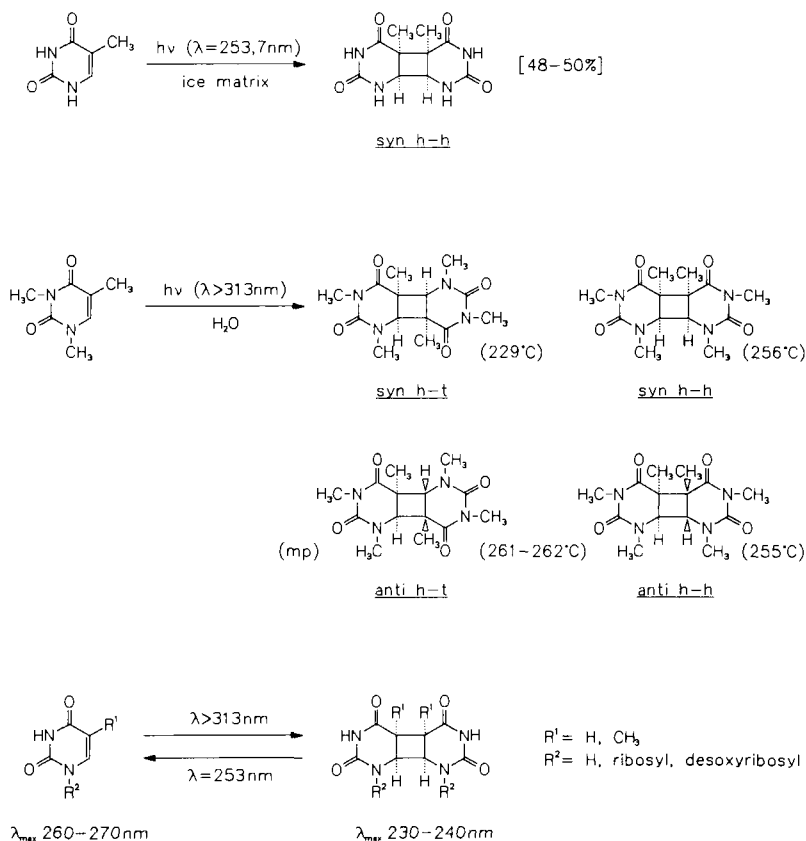
SCHEME 21. Wavelengths of absorption (extinction) of some nucleic bases and nucleosides [λ_{max} (nm) (lg ϵ)].

A second important photoreaction of uracils observed in aqueous solution is the photochemical addition of one molecule of water to form 6-hydroxy-5,6-dihydro uracils (55SCI594; 56JA4180; 58JA6196; 70T5913; 77TL1661). The irradiation of 1,3-dimethyluracil in methanol leads to the formation of four different products. The product distribution is very sensitive to the wavelength used [75TL477; 76MI2; 77TL3397; 84TL(25)1521]. Use of EuCl_3 as a catalyst affords predominantly 6-hydroxymethyluracils (85CC1481). Intramolecular addition of 5-hydroxyalkyl-4-thiouracils proceeds to furnish the corresponding 5,6-cyclic 5,6-dihydrouracils (76TL2375) (Scheme 23).

Upon UV irradiation, thymine-1-yl-acetic acid shows decarboxylation that is competitive with dimerization (68CC1162 (Scheme 24).

In the presence of benzophenone sensitizer, dimerization is observed accompanied by oxetane formation (67MI1). UV irradiation of 6-acetyluracil in water gives a tetracyclic oxetane (79MI1; 83MI2) (Scheme 25).

Several dinucleotide model compounds have also been used in photo experiments. These intramolecular photodimerizations are controlled through the "spacer" groups (73JA2320; 74BAP393; 74JA5904; 78MI2, 78MI3; 84H1363) (Scheme 26).

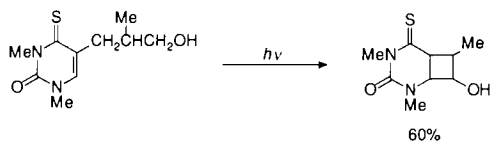
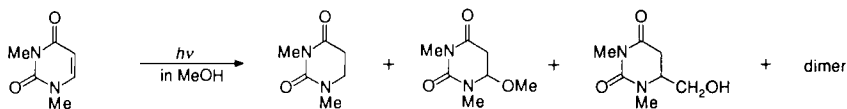
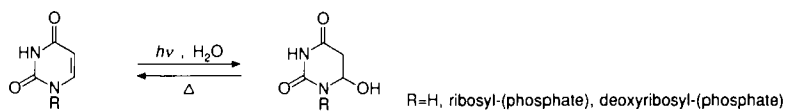


SCHEME 22

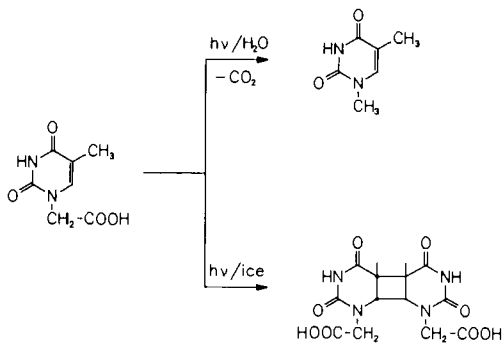
Using 1,1'-linked uracils, intramolecular dimerization is wavelength dependent and also reversible, as shown in Scheme 27, (88CJC1027). Such a photoreversible reaction provides a rare example of a C=C to C=N [2 + 2]-photocycloaddition (76TL449) (Scheme 27).

Along with dimerization and intramolecular cycloaddition, photocycloaddition of several other molecules to the 5,6-double bond of uracils is significant.

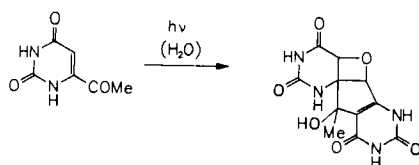
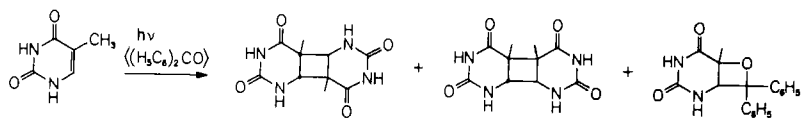
- (1) Alkenes (71SCI435; 72MI1) add to 6-azauracils (74JA4879), including photoaddition of terminal olefins (76JA1602; 80JOC4462; 82TL2571; 83TL4055) (Scheme 28).
- (2) Ketene acetals (72JA7605; 74JA4885; 88JOC1530) reactants are shown in Scheme 29.



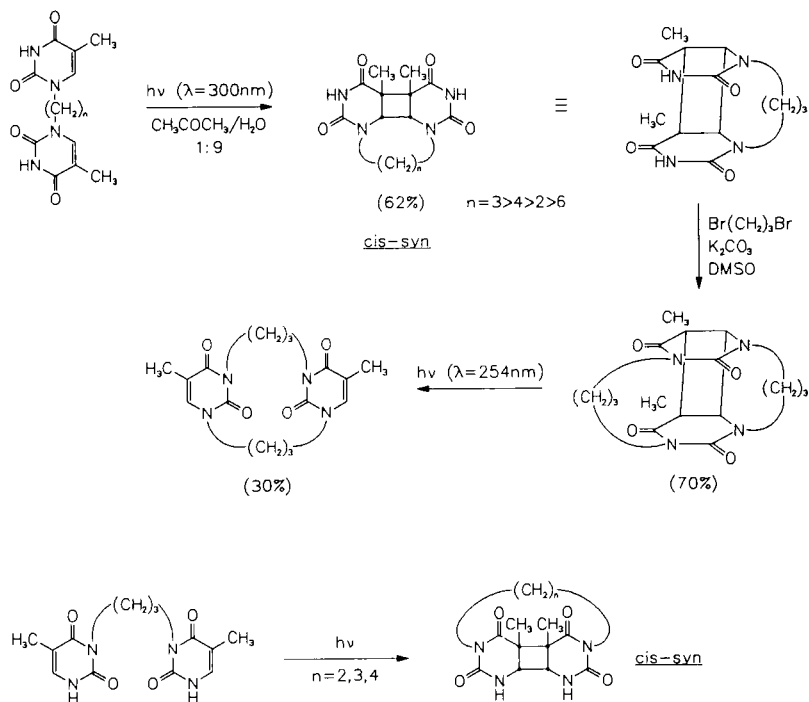
SCHEME 23



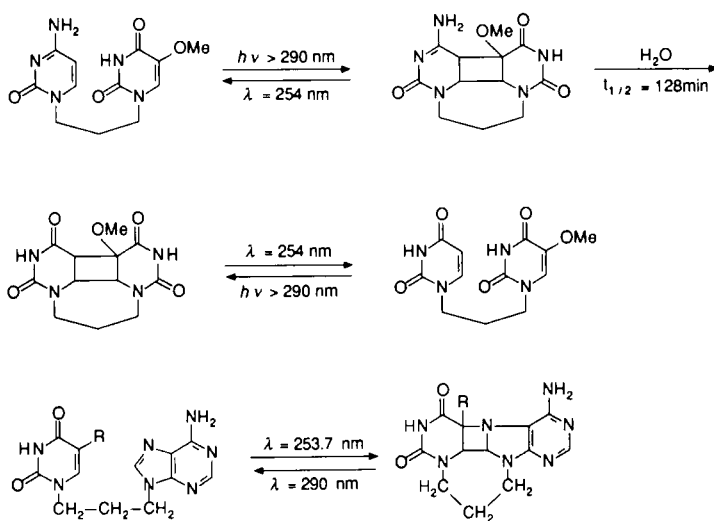
SCHEME 24



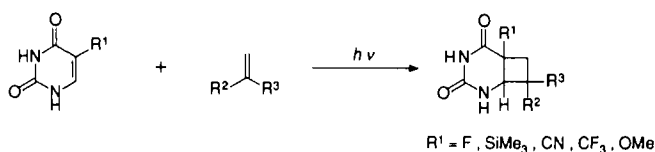
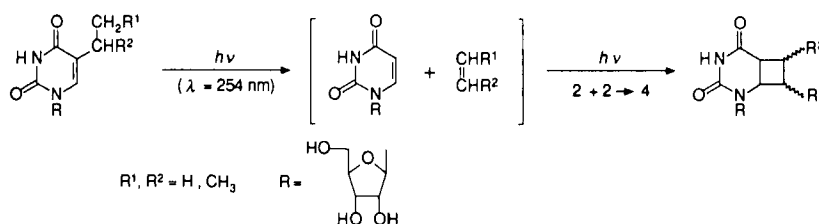
SCHEME 25



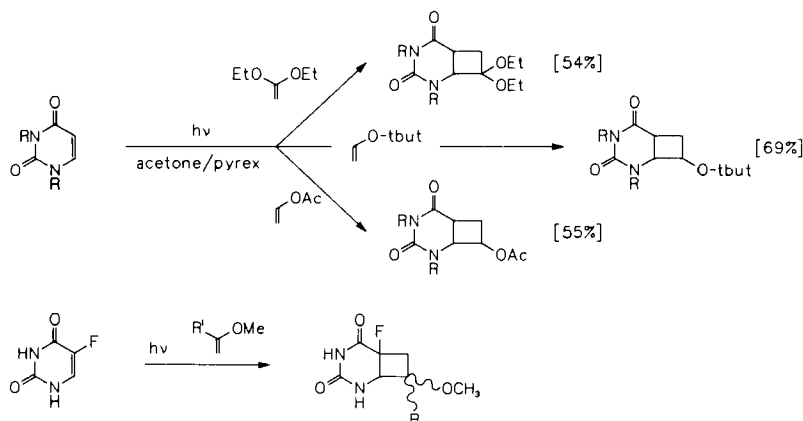
SCHEME 26



SCHEME 27



SCHEME 28

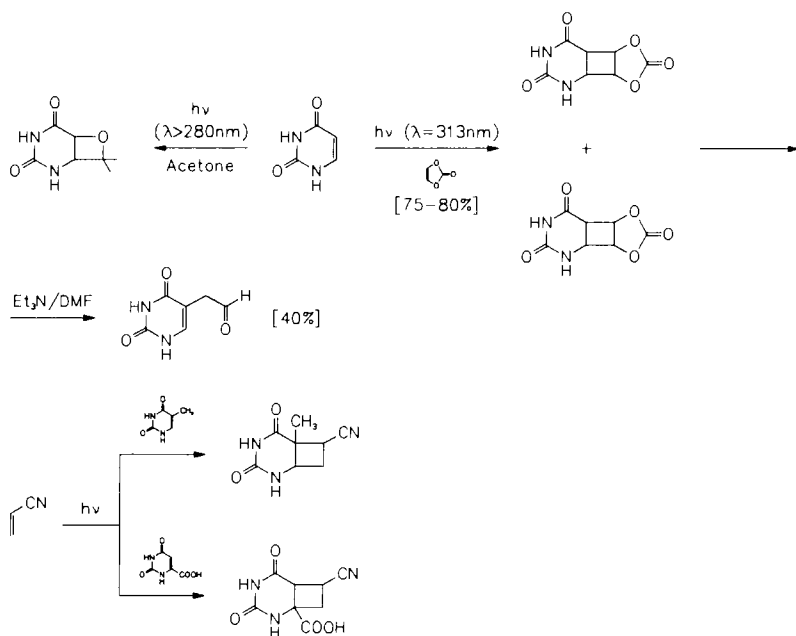


SCHEME 29

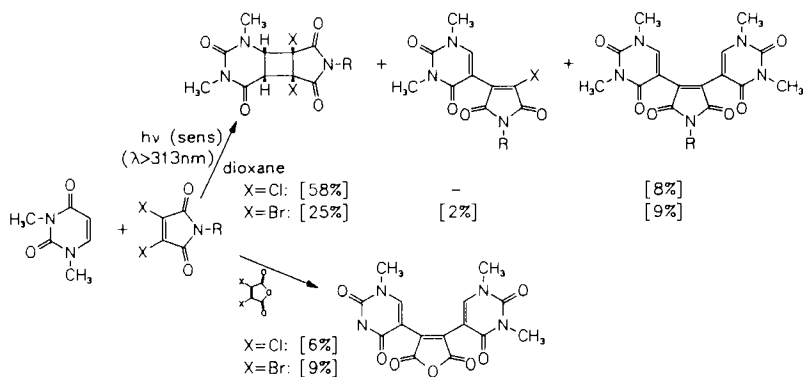
(3) Ketones and vinylene carbonates [72CRC(C)882; 74TL1087; 75MI1; 79JA6398] and acrylonitril (70MI1) react as indicated in Scheme 30.

Dihalogenmaleinimides (DBMI) have been found to be powerful photocyclophiles toward uracils [80AG1066, 80AG(E)1026, 80CB(92)1066], including 6-azauracils (Scheme 31).

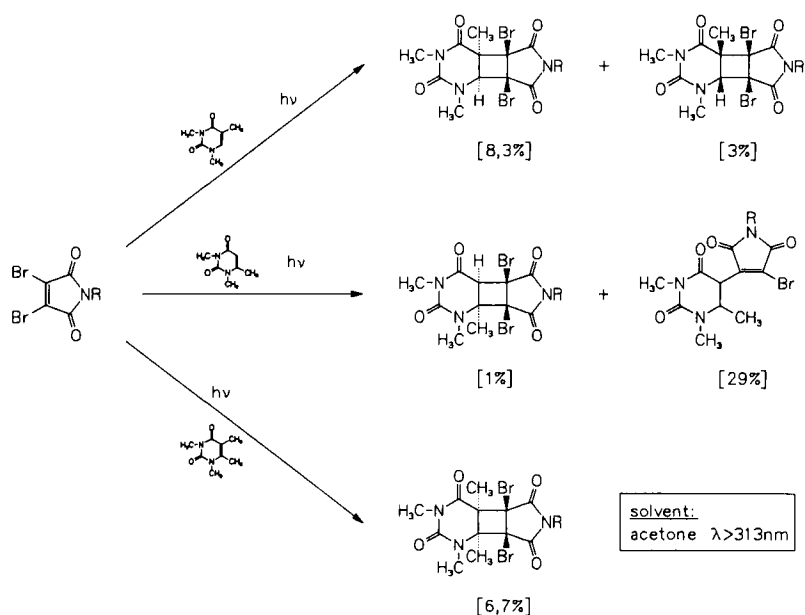
In addition, substituent effects on the uracil ring have been investigated in photocycloadditions with DBMI [83AG156, 83AG(E)157, 83AG(S)120] (Scheme 32).



SCHEME 30

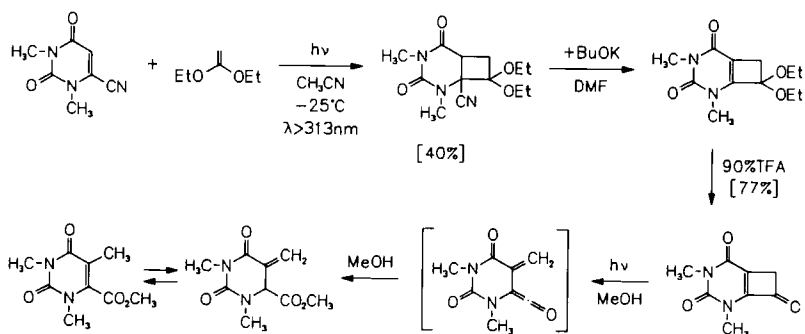


SCHEME 31



SCHEME 32

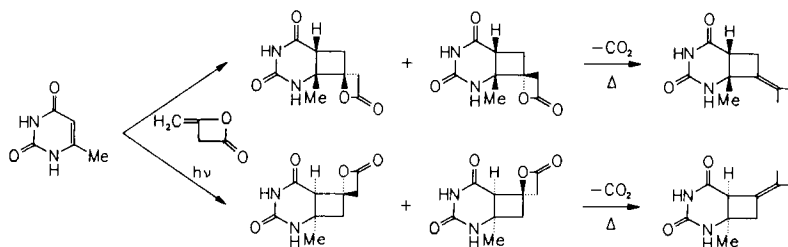
The key step in the synthesis of 2,4-dimethyl-2,4-diazabicyclo[4.2.0]oct-1(6)-ene-3,5,8-trione is a photocycloaddition of a ketene acetal [83AG639, 83AG(E)629, 83AG(S)835] (Scheme 33).



SCHEME 33

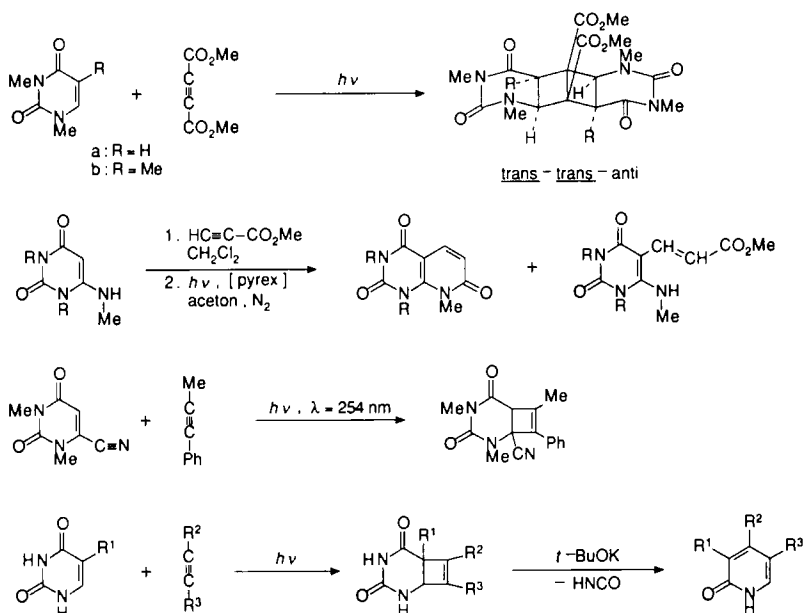
Upon UV irradiation, 6-methyluracil undergoes [2 + 2]-cycloaddition with a diketene to give spirooxetanones. The four isomeric diazabicyclo[4.2.0]octane-spirooxetanones are thermally decarboxylated to

afford two exomethylene 2,4-diazabicyclo[4.2.0]octanes (82CPB544) (Scheme 34).



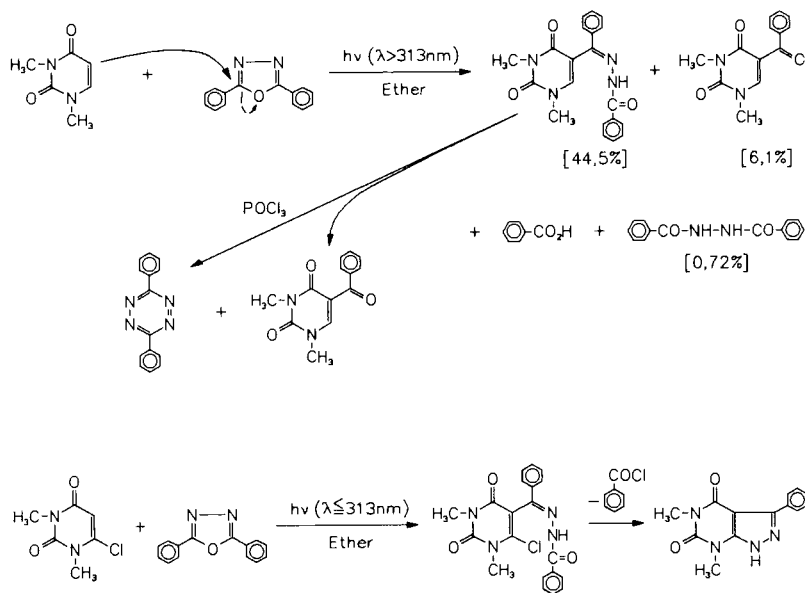
SCHEME 34

Dimethyl acetylenedicarboxylate reacts, upon UV irradiation, with 1,3-dimethyl-uracil in a [2 + 2]-cycloaddition (79H1175), while 6-aminouracils and propiolic esters both photochemically and thermally give pyrido[2,3-*d*]pyrimidine and the preceding Michael adduct (87JHC1453). Accordingly, 6-cyano-1,3-dimethyluracil and methylphenylacetylene give, by reversible [2 + 2]-photocycloaddition, a cyclobutene derivative (80TL2317). The uracil-alkyne photoadduct rearranges to 2-pyridones upon treatment with 2 equivalents of potassium *tert*-butoxide (83JOC2337) (Scheme 35).



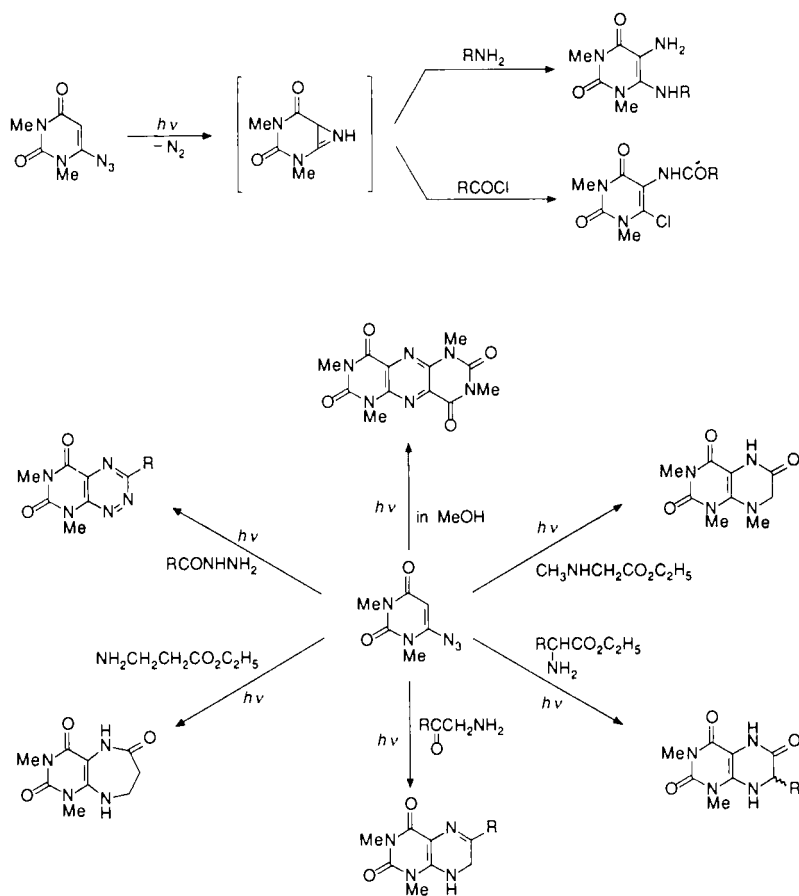
SCHEME 35

When 1,3-dimethyluracil is irradiated in the presence of 1,3,4-oxadiazoles, no [2 + 2]-cycloadduct is formed (68TL3971; 73H1101, 73T41; 76CL153; 77BCJ3281). Instead, a ring-cleaving cycloaddition takes place to form acylated hydrazones and side products [80CB(113)2556]. This photoreaction can be used as an easy and selective 5-benzoylation procedure. In the case of 6-chloro-1,3-dimethyluracil, the intermediate hydrazone is photochemically cyclized to a pyrazolo[3,4-*d*]pyrimidine [80CB(113)2566] (Scheme 36).



SCHEME 36

Photodecomposition of 6-azido-1,3-dimethyluracil in the presence of alkylamines and acyl chlorides can be used as a convenient method of preparing 6-alkyl-amino-5-aminouracils and 5-acylamino-6-chlorouracils, respectively (76CC731; 78CC367). The former reaction was applied to the synthesis of a large variety of heterocycle-fused uracils. In the presence of methanol, a pyrimido[4,5-*g*]pteridinetetrone results via a nitrene intermediate (58LA57). In the presence of ethyl *N*-methylglycinate, α -amino-carbocyclic esters and -ketones, acylhydrazine or ethyl- β -alanine ethyl ester, 7,8-dihydrolumazine, its 6- and 7-substituted derivatives, fervenuline and pyrido[4,5-*b*][5,9]diazepine, respectively, are easily accessible (77JA7358; 78JA7661) (Scheme 37).



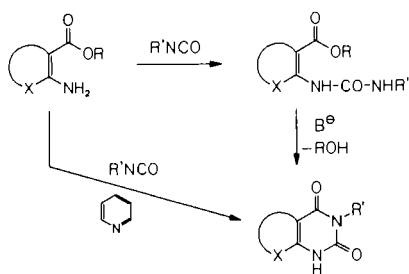
SCHEME 37

VII. Annulation Reactions to the Intact Uracil Molecule

Several bi- and oligocycles containing uracil rings were found to be an important class of compounds that often possess interesting pharmacological activity. These compounds include antiviral and anticancer agents as well as crop protecting materials (69AHC149; 82MI1; 83MI1; 84MI1; 85AHC299; 86CZ425).

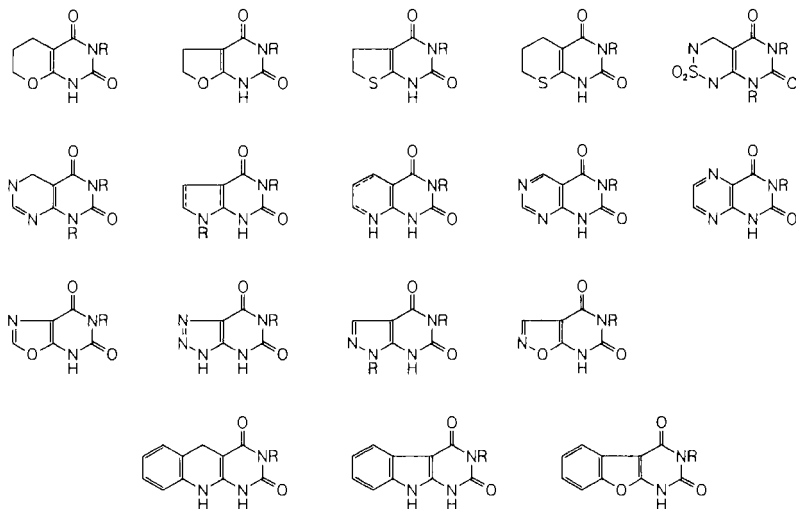
A. HETEROCONDENSED URACILS FROM LACTONES AND HETEROCYCLIC β -ENAMINO ESTERS

The treatment of heterocyclic and heteroaromatic β -enamino esters with isocyanates followed by ring closure with a suitable base is a general synthetic route that leads to bi- and triheterocondensed uracils in reasonable yields. Starting materials are easily accessible (68CB3377; 73CB3533, 73S546; 74CB2265; 75S426; 76CB2983; 78CB2297; 80MI2; 85AHC299) Scheme 38).



SCHEME 38

By this means, a large number of heterocondensed uracils have been obtained; some are depicted in Scheme 39.



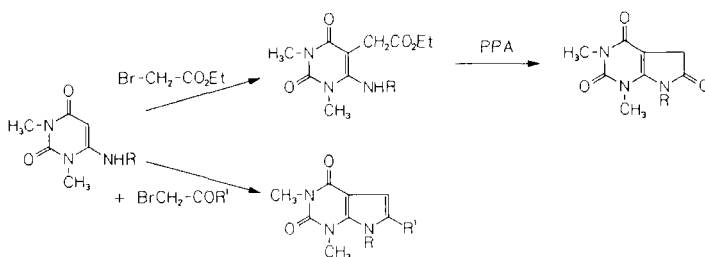
SCHEME 39

As mentioned, these heterocondensed uracils are unsubstituted in position 1, so subsequent nucleosidation leads to unusual nucleosides [91JPR(ip)].

B. 6-AMINO-1,3-DIMETHYLURACILS

Uracil and 1,3-dimethyluracil, the title compounds of this review, are not well suited for the preparation of additional heterocyclic rings. However, 5- and 6-substituted derivatives are more promising.

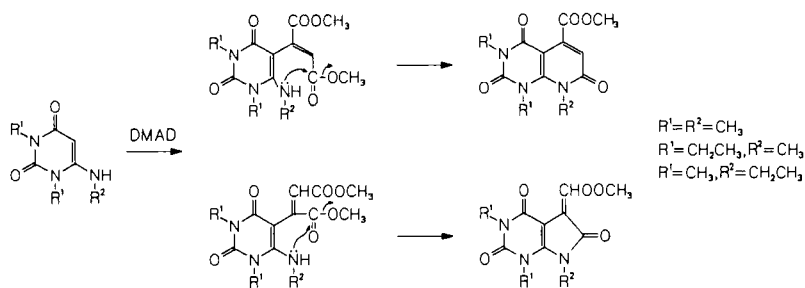
Thus, 6-amino-1,3-dimethyluracils are alkylated in the electron-rich 5-position, and the ester intermediate cyclizes at 140–150°C in the presence of PPA to afford pyrrolo[2,3-*d*]pyrimidines; α -haloketones react accordingly (64JHC34; 72CPB404; 73CPB473; 74CPB1459) (Scheme 40).



SCHEME 40

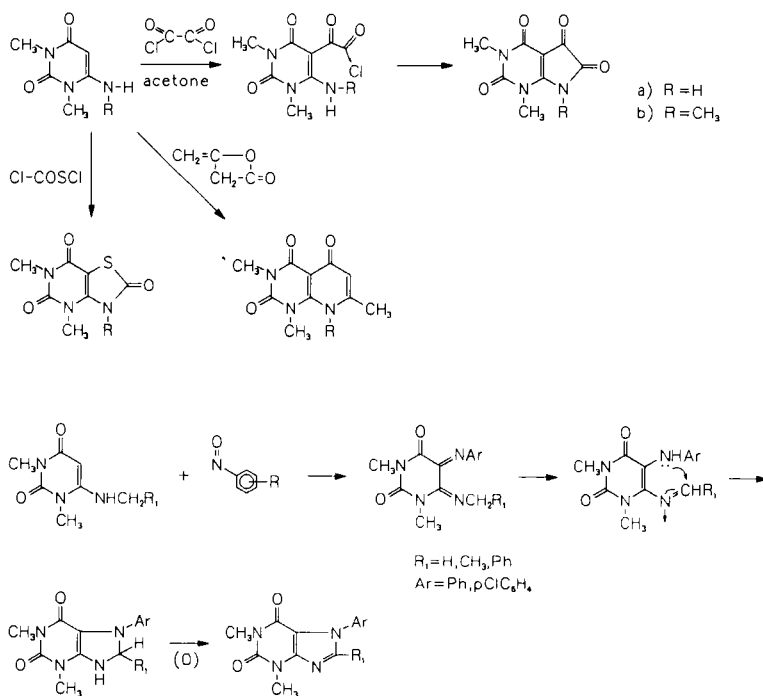
Based on earlier publications by Broom *et al.* (72JOC578; 76JOC1095; 77JOC4159), Ogura and Sakaguchi reported on the products formed by treatment of 6-aminouracils with dimethyl acetylenedicarboxylates (DMAD) (73CPB2014). Besides acylation reactions on the 5-position, a Michael adduct was found to lead to pyrido[2,3-*d*]pyrimidines and, after heating to 165–175°C, to pyrrolo[2,3-*d*]pyrimidine (82CPB63; 86H927; 87JHC1215). These results, however, may not be correct and may be revised in the light of additional findings discussed later (86JOC149, 86JOC2787; 89CB1673). Analogous reaction of dibenzoyl ethylene in place of DMAD gives also pyrido[2,3-*d*]pyrimidines or pyrrolo[2,3-*d*]pyrimidines or both (75H183) (Scheme 41).

Furthermore, 6-aminouracils are attacked by oxalyl chloride, diketene, and chlorocarbonylsulfenyl chloride at C-5, followed by cyclization with the amino group to give pyrrolo[2,3-*d*]pyrimidines (79JHC717), pyrido[2,3-*d*]pyrimidines (73CPB2014), and thiazolo[4,5-*d*]pyrimidines (73LA1018), respectively. 6-Amino-1,3-dimethyluracil and nitrosoben-



SCHEME 41

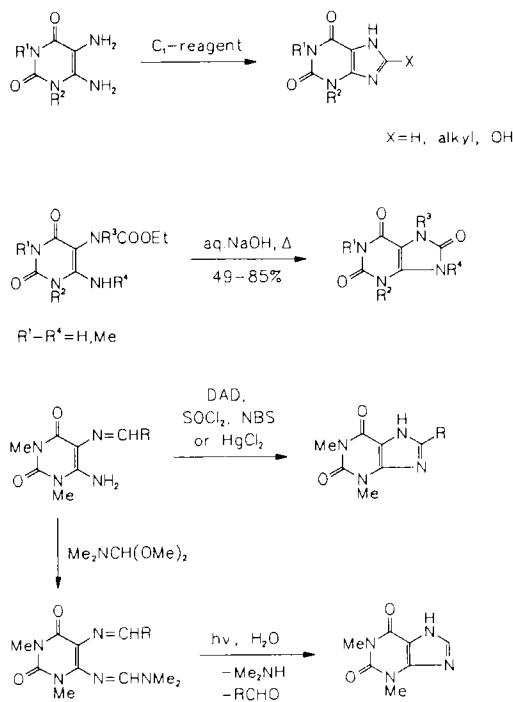
zene in the presence of acetic anhydride condense to 7-phenyltheophylline via a diimine intermediate (72JOC4464) (Scheme 42).



SCHEME 42

The Traube purine synthesis using 5,6-diaminopyrimidines is well known (1900CB1371, 1900CB3035). According to this method, uric acids are formed from 5,6-diaminouracils and one carbon (C1) reagents, such as

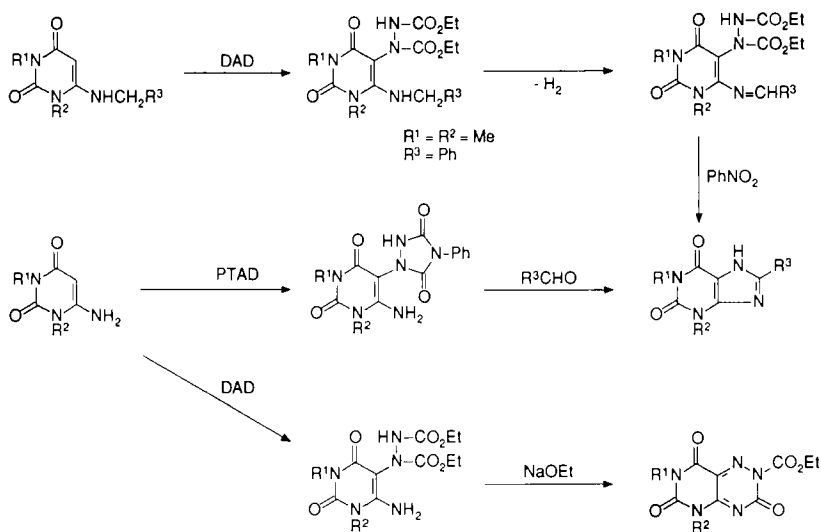
formic acid, ethyl chloro-formate, and formamide (71CHE(24)31]. Analogous treatment with thioisocyanates leads to the formation of 8-aminoxanthine derivatives (79CPB1153). Furthermore, the alkaline cyclization of 6-aminouracil-5-carbamates gives uric acids (74LA2030), while oxidative cyclization of 6-amino-5-benzylideneaminouracil in the presence of nitrobenzene (66AG679), diethyl azodicarboxylate (DAD) [76H(4)1759; 78CPB2905], mercury(II)chloride (79CPB1094, 79H359), *N*-bromosuccinimide (NBS) [77H(6)1919], or thionyl chloride (77CPB495; 78CPB3240) gives xanthine derivatives. When dianils of 5,6-diamino-1,3-dimethyluracil are exposed to sunlight, a 1,5-ring closure reaction occurs to give theophylline [76H(4)1659] (Scheme 43).



SCHEME 43

Reaction of benzylaminouracil with DAD affords xanthines via a Michael addition and internal disproportionation reaction [75CC146; 77JCS(P1)1754]. The Michael adduct of 4-phenyl-1,2,4-triazoline-3,5-dione (PTAD) is easily converted into xanthines on heating with aromatic aldehydes [74CC551; 77JCS(P1)2285]. Insoferevenulin can be pre-

pared by treatment of the DAD adduct with sodium ethoxide (75JOC2329) (Scheme 44).



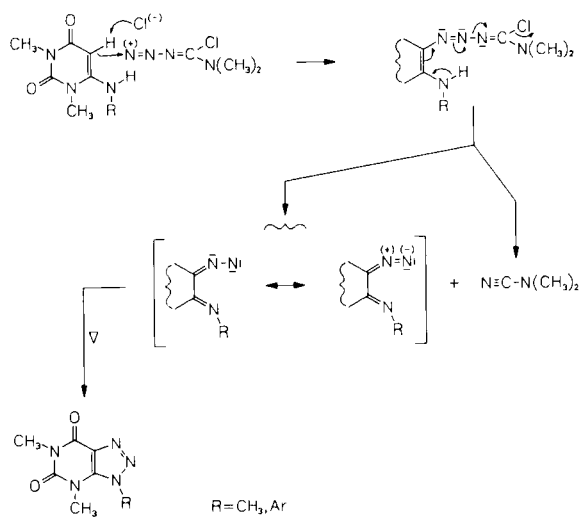
SCHEME 44

8-Azatheophyllines are formed in high yield in a one-step synthesis from 6-aminouracils and *N,N*-dimethylazidochloromethyleneiminium chloride via diazo group transfer in a one-pot reaction. A metastable triazine functions as an intermediate which, after rearrangement and thermal ring closure, gives 1,2,3-triazolo[4,5-*d*]pyrimidines (8-azatheophyllines) (87JHC1493) (Scheme 45).

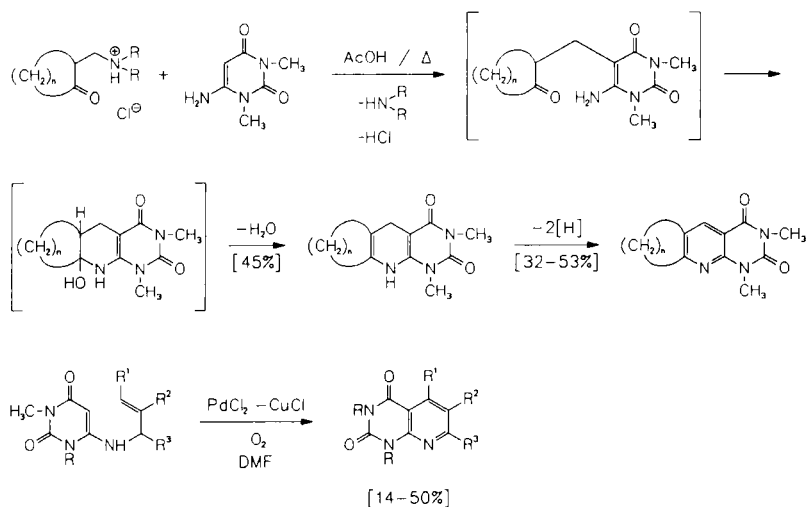
Cycloalka[*g*]pyrido[2,3-*d*]pyrimidines can be made via a Mannich reaction from 6-amino-1,3-dimethyluracil and aminomethylated cycloketones (78AP406, 78AP542). A simple approach to pyrido[2,3-*d*]pyrimidines consists of a Pd-catalyzed cyclization of 6-allylaminouracils (83H2177). An improved procedure was reported for synthesizing pyrido- and pyrrolo[2,3-*d*]pyrimidines from allylaminouracils with PdCl_2 at 60°C and the ribosides thereof (89CPB3184) (Scheme 46).

Pyrido[2,3-*d*]pyrimidines are biological, highly active compounds possessing antitumor, antibacterial, and anticonvulsive activities (80CPB761, 80JMC327, 80PHA253).

Pyrido[2,3-*d*]pyrimidines are easily constructed from 6-aminouracils and acroleine [92MI1(up)], α,β -unsaturated ketones [72JCS(P1)1041; 74JCS(P1)1225; 76JOC3149; 81IJC(B)159; 88JCS(P1)2993], or methylene

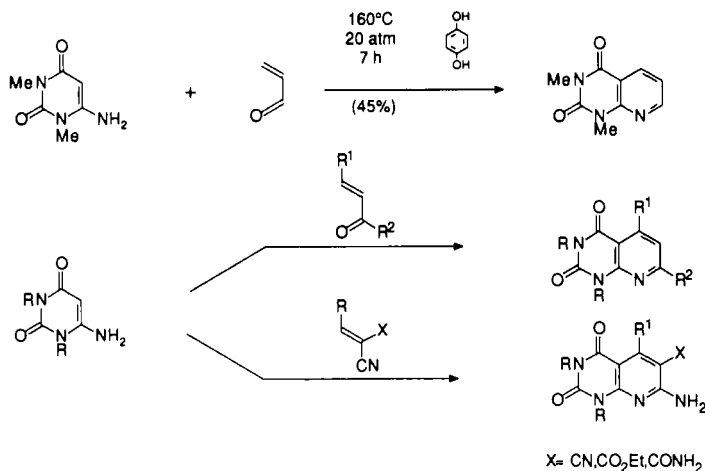


SCHEME 45



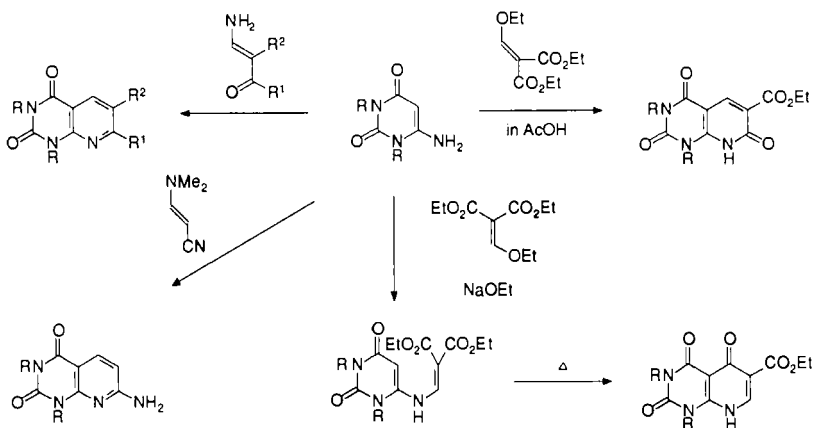
SCHEME 46

malononitriles (84CC1549; 90JOC568) via Michael addition at position C-5 on the uracil ring (Scheme 47).



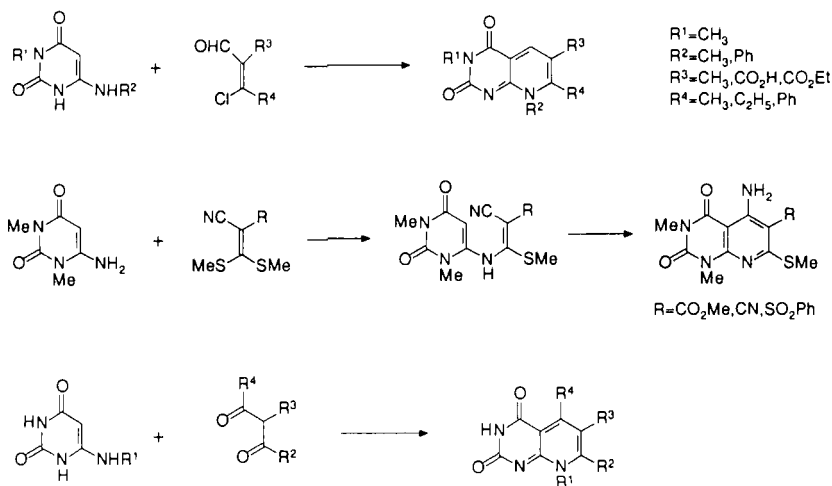
SCHEME 47

An analogous method for synthesizing pyrido[2,3-*d*]pyrimidines is achieved using activated olefinic systems possessing a leaving group, such as dimethylamino or ethoxy (73SC397, 73T2209; 74CB2537). When diethyl ethoxymethylene-malonate is used under acidic conditions, 7-oxopyrido[2,3-*d*]pyrimidines are formed. But in the presence of sodium ethoxide, the Michael addition of the 6-amino group preferentially proceeds as described in Scheme 49 to give 5-oxo-pyrido[2,3-*d*]pyrimidines (85JHC1469) (Scheme 48).



SCHEME 48

A versatile synthesis of 5-desazalumazines consists of a substitution and condensation reaction of chloroalkenates to aminouracils (83S75; 84CPB1699). Similar reaction with 3,3-bis(methylthio)acrylonitriles gives 5,6,7-trisubstituted pyrido[2,3-*d*]pyrimidines (79H503; 84CPB122), which are formed by condensation of 6-aminouracils with 1,3-diketones [72JCS(P1)1041; 75JHC1221] (Scheme 49).



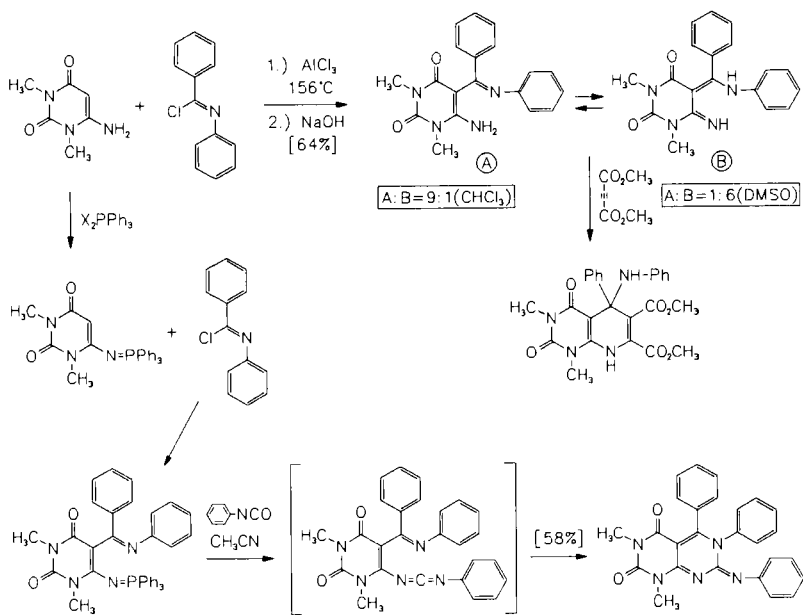
SCHEME 49

In a more recent approach, 6-aminouracil or its 6-iminophosphorane is reacted with *N*-phenylbenzimidoyl chloride to give, in a Michael-type addition, a tautomeric pair of imines, A and B. The equilibrium is shifted in different solvents. Tautomer B is intercepted by an acetylenic ester to form a 1,4-dihydropyrido[2,3-*d*]pyrimidine. The iminophosphorane of A and isocyanate results in an aza-Wittig reaction and polar 6 π -electron cyclization reaction to give pyrimido[4,5-*d*]pyrimidines [91TL(ip)]6 (Scheme 50).

Similarly, pyrido[2,3-*d*]pyrimidines are formed from the same 6-iminophosphorane; 1-dimethylamino-2-nitroethylene followed by aryl isocyanates in a so called tandem aza-Wittig/electro cyclization strategy (90S474) (Scheme 51).

Diazahexatriene systems generated on a uracil spacer molecule are cyclized to give 6-aryllumazines [76H(4)977, 76H(4)1659; 77JCS(P1)1336]. Another approach to a lumazine is accomplished by oxidative cyclization of 6-amino-5-hydroxyethylidene-aminouracils with mercury(II)chloride (79CPB1094, 79H359) (Scheme 52).

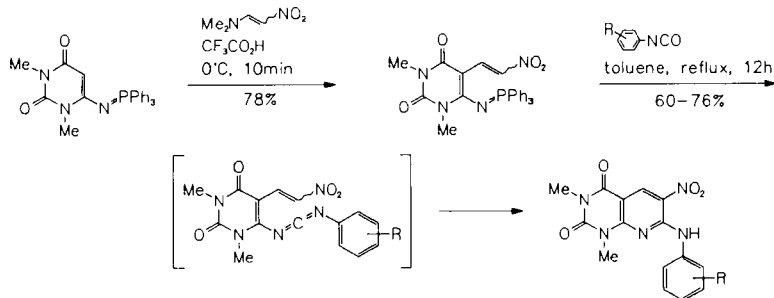
5,6-Diaminouracils are rather versatile starting materials for a number of interesting pteridine derivatives [88CHE(24)44]. Reaction partners are



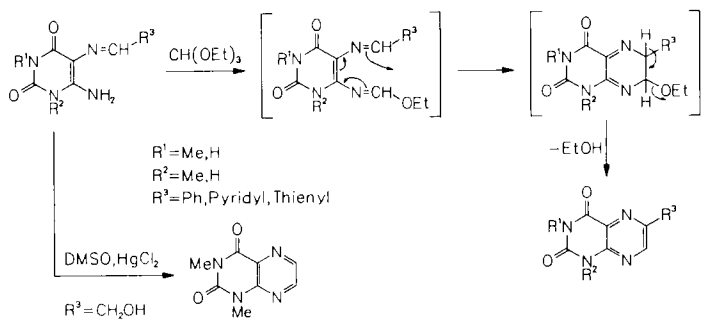
SCHEME 50

α -keto-propionaldehyde, α -oximinoacetone (87JHC597), ethoxyiminoacetate (82TL3357) or trifluorhaloacetone (88JOC5088), and benzylidene pyruvic acid. The latter reagent leads to 7-hydroxy-6-styryl- and to 7(2-arylvinyl)-6-hydroxypteridine (87JHC1587) (Scheme 53).

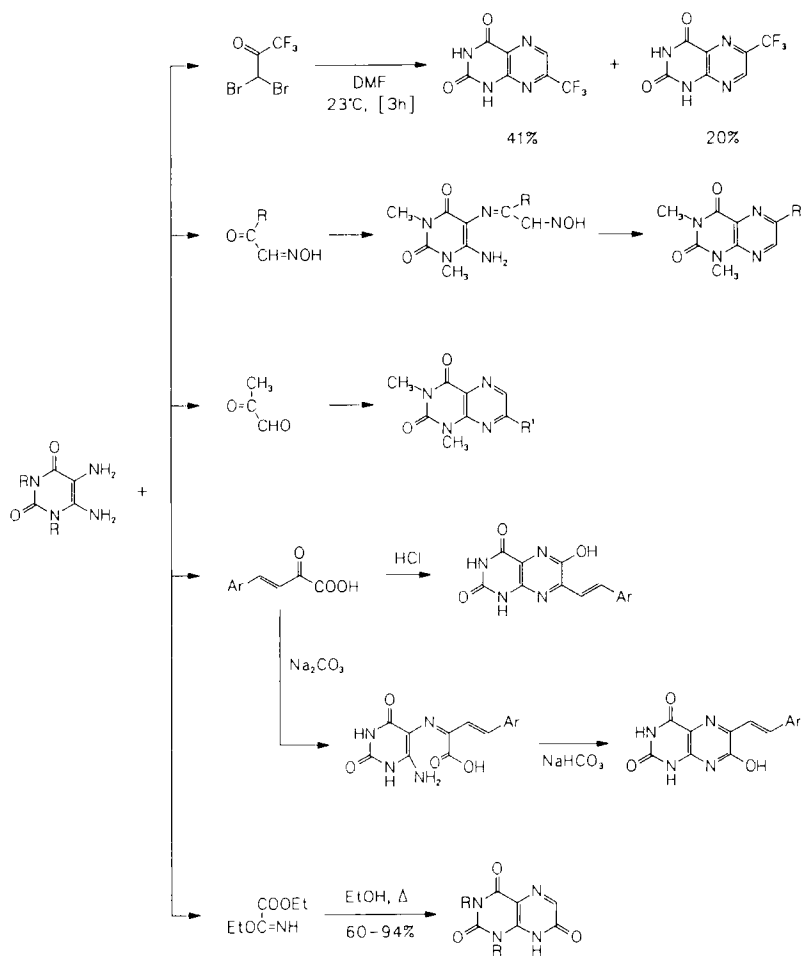
1,2,3-Triazolo[4,5-*d*]pyrimidines and 1,2,5-thiadiazolo[3,4-*d*]pyrimidines are derived from the reaction of 5,6-diaminouracils with sodium nitrate and thionyl chloride, respectively (54JA2798; 65CB1060; 74MI1; 78H1437; 89MI16) Scheme 54).



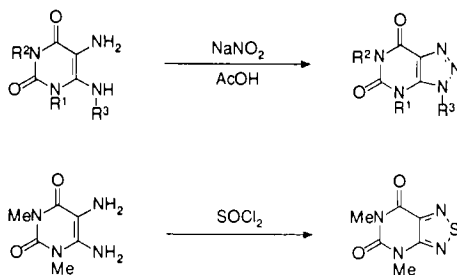
SCHEME 51



SCHEME 52

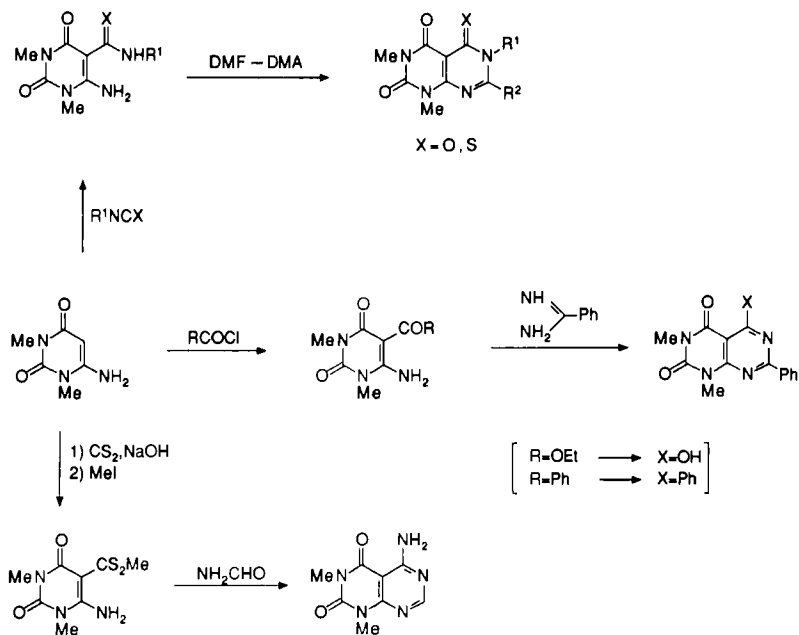


SCHEME 53



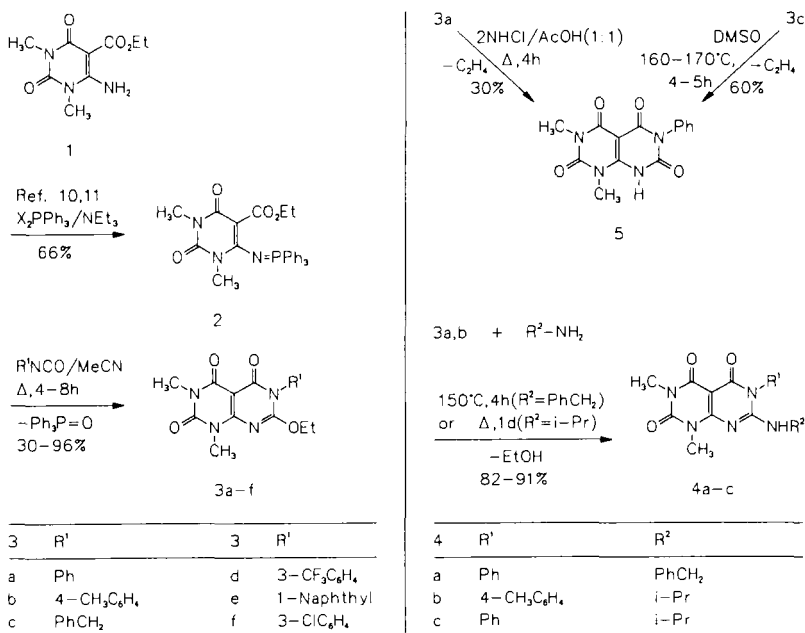
SCHEME 54

Next to pyrido[2,3-*d*]pyrimidines, pyrimido[4,5-*d*]pyrimidines belong to a class of compounds of biological relevance, both in the plant protection area and especially as plant growth regulators, because of their connection with purine and pteridine systems. Simple access to this class is found in the reaction of 6-amino-5-carbamoyluracil with typical one-carbon reagents, such as dimethylformamide (DMF) dimethylacetal, acylanhydrides, and *N,N'*-carbonyldiimidazole (77MI1; 86H2293). 6-Amino-5-ethoxycarbonyl (or benzoyl)uracils and 6-aminouracil-5-carbodithionate react with benzamide and formamide, respectively, to give pyrimido[4,5-*d*]pyrimidines (73BCJ3849; 79CL155, 79H503, 79YZ515) (Scheme 55).



SCHEME 55

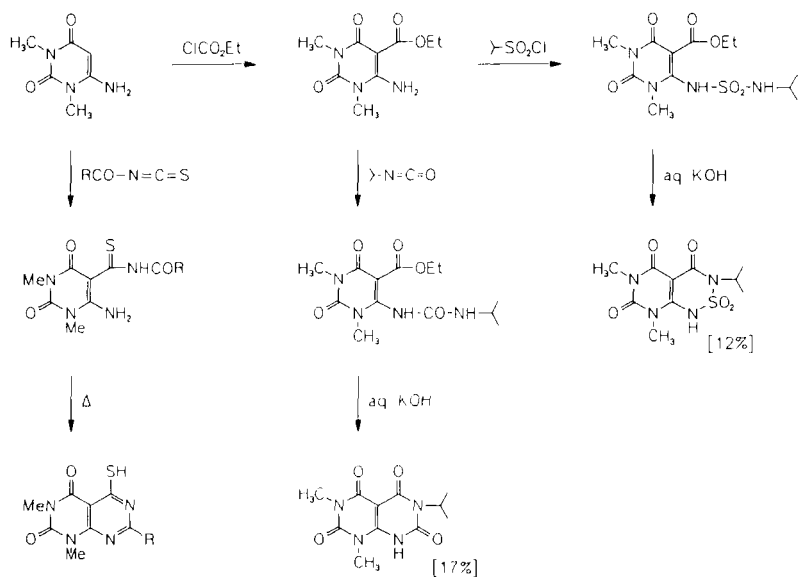
Accordingly, uracil- β -enamino ester, after treatment with dihalotriphenylphosphorane (via iminophosphorane), gives, with isocyanates in a normal kind of pyrimidine annulation (90LA901), pyrimido[4,5-*d*]pyrimidines containing a 2-alkoxy group capable of several subsequent reactions (88S919) (Scheme 56).



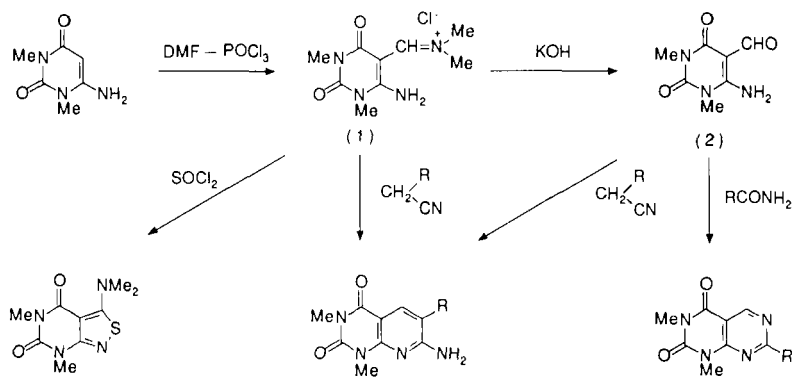
SCHEME 56

Reaction with isocyanates and isopropyl-sulfonyl-chloride, via the uracil-enamino esters, easily gives pyrimido[4,5-*d*]pyrimidines and pyrimido[4,5-*d*][2,1,3]thiadiazines (87UP1). Thermal cyclization of 6-amino-uracil-5-thiocarboxamides, prepared by reacting 6-aminouracils with isothiocyanates yields pyrimido[4,5-*d*]pyrimidines (70JHC243; 74LA2019) (Scheme 57).

Vilsmeier reaction of 6-amino-1,3-dimethyluracil using DMF-POCl₃ gives a stable intermediate (1) which is easily hydrolyzed to the corresponding 5-formyluracil (2). Both compounds 1 and 2 condense with various active methylene compounds to afford pyrido[2,3-*d*]pyrimidines. Condensation of 2 with acid amides provides a convenient synthetic method for producing pyrimido[4,5-*d*]pyrimidines (68CB512; 80BSB651; 83KGS834; 84LA1653, 84S589; 85JHC345). Isothiazolo[3,4-*d*]pyrimidines are formed from the intermediate (1) and thionyl chloride (76CPB970) (Scheme 58).

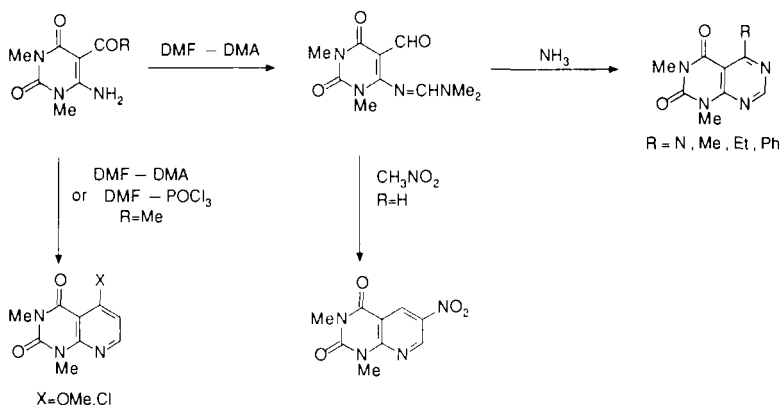


SCHEME 57



SCHEME 58

5-Substituted pyrimido[4,5-*d*]pyrimidines are synthesized by reaction of 5-acyl-6-aminouracils with dimethylformamide dimethylacetal (DMF-DMA) and subsequent cyclization by ammonia. When nitromethane is used instead of ammonia, 6-nitropyrido[2,3-*d*]pyrimidine is formed, starting from the 5-formyluracil. 5-Methoxy- and 5-chloropyrido[2,3-*d*]pyrimidines are obtained by direct cyclization of 5-acetyl-6-aminouracil using DMF-DMA and DMF-POCl₃, respectively, under drastic conditions [91JMC(ip)] (Scheme 59).



SCHEME 59

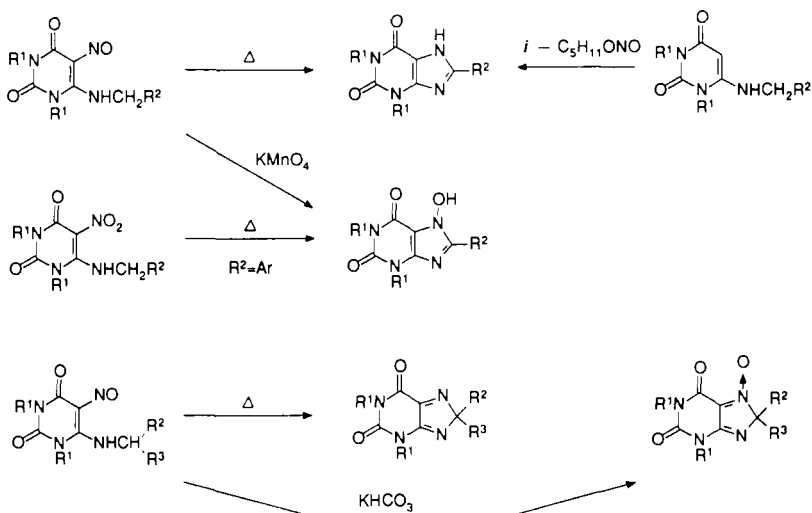
Chloro substituted pyrimido[4,5-*d*]pyrimidines are obtained by cycloaddition of 6-aminouracil with (perchloroalkylideno)-polychloroalkylamines and perchloroethyl isocyanate; the 1,3,5,7-substituted derivatives are related to the 2,4,7-trisubstituted pyrimido[4,5-*d*]pyrimidines (74LA2066) (Scheme 60).

Pyrimido[5,4-*b*][1,4]thiazines are synthesized by reaction of 6-amino-5-chloro-uracils with mercaptoacetic acid in alkaline solution, followed by dehydration in acetic anhydride (62JA1904). Chlorosulfonation and subsequent amination of 6-aminouracil give the corresponding 5-sulfonamides, which are cyclized to pyrimido[4,5-*e*][1,2,4]thiadiazines on heating in triethyl orthoformate (63JOC1994) (Scheme 61).

Another simple way to access 3-aminoisothiazolo[3,4-*d*]pyrimidines is oxidative cyclization of *o*-aminodithiocarboxylates (76CPB979; 79H485) or 6-aminouracil-5-thiocarboxamides (74LA2019) with halogens. Glucosyl isothiocyanates lead directly to nucleosides (79CPB1147) (Scheme 62).

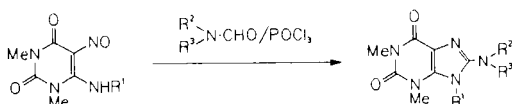
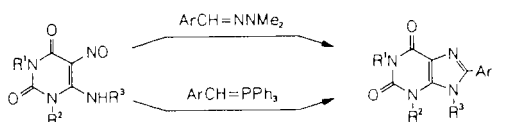
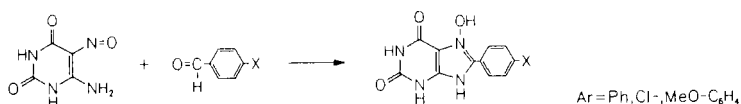
C. AMINO/NITROSO- AND AMINO/AZOURACILS

Another versatile set of substituents for heterocyclization is a combination of 6-amino and 5-nitroso (or azo) groups: thus, 6-amino-5-nitroso (or azo) uracils are good starting compounds for the synthesis of xanthine derivatives [71CHE(24)72]. 8-Substituted xanthines are conveniently synthesized by heating of 6-alkyl (or aralkyl)amino-5-nitrosouracils or by nitrosation of 6-alkylaminouracils with isoamyl nitrite [66LA(691)142, 66(698)LA145, 66(699)LA145; 87CCC2730]. On the other hand, reaction of the 5-nitrosouracils with isoamyl nitrite or potassium permanganate gives 7-hydroxyxanthines, which are also obtained by heating 6-benzylamino-5-nitrouracils [84JCS(P1)583]. When 5-nitrosouracils bearing an α -carbon-branched alkylamino group are used as starting materials, 8,8-dialkylxanthines and their 7-oxides are formed (65TL2701; 66LA134) (Scheme 63).



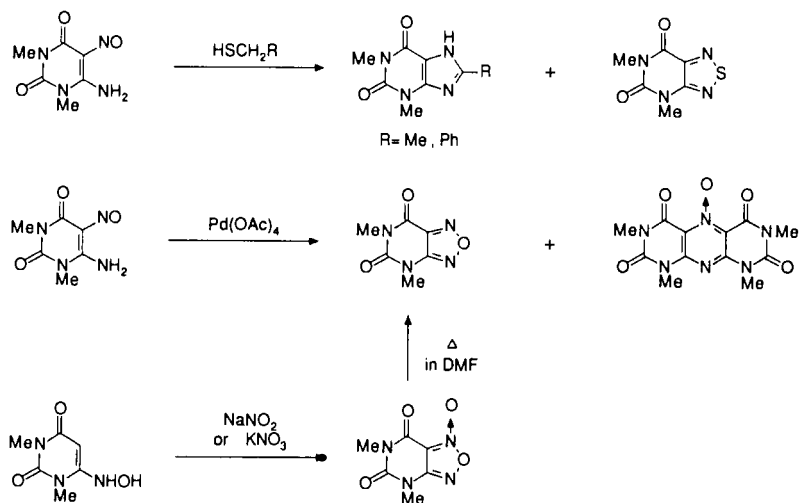
SCHEME 63

6-Amino-5-nitrosouracils react with benzaldehydes to afford 7-hydroxyxanthines (82JHC205). On the other hand, the reaction with benzaldehyde N,N -dimethyl-hydrazones or Wittig reagents (benzylidenetriphenylphosphoranes) gives 8-aryl-xanthines [76CC155, 76JCS(P1)1547]. 8-(Substituted amino)xanthines are formed by treatment with substituted formamides-phosphorus oxychloride (a kind of Vilsmeier reagent) (72CC606; 73BCJ1836; 74CPB1658) (Scheme 64).



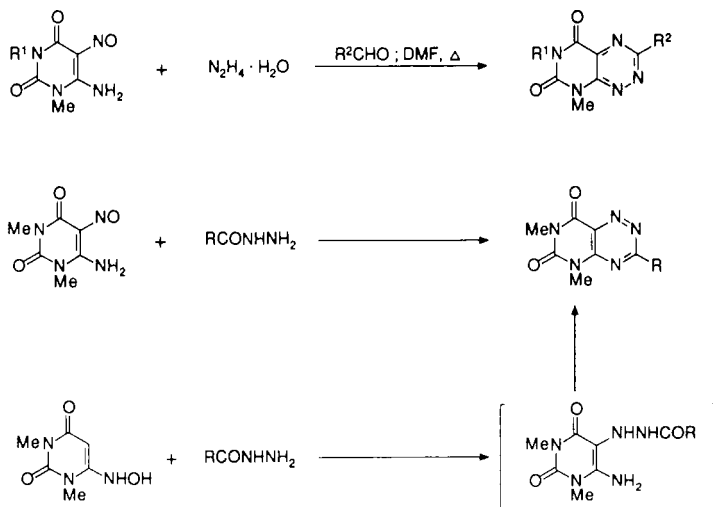
SCHEME 64

Condensation of 6-amino-5-nitrosouracils with ethanethiol and phenylmethanethiol leads to formation of 8-substituted xanthines and 1,2,5-thiadiazolo[3,4-*d*]pyrimidines [75JCS(P1)1857]. Oxidation with lead tetraacetate forms furazano[3,4-*d*]pyrimidine and pyrimido[5,4-*g*]pteridine 10-oxide (72JOC1601). The former product is also prepared by reaction of 6-hydroxyuracil with sodium nitrite or potassium nitrate and subsequent heating in DMF [73JHC415, 73JHC993; 76JCS(P1)1327] (Scheme 65).



SCHEME 65

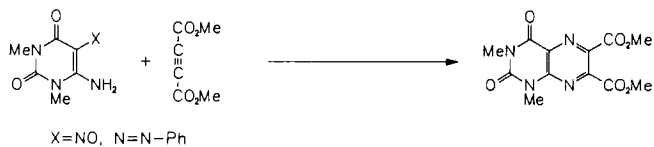
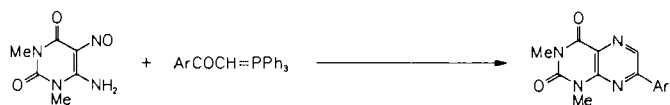
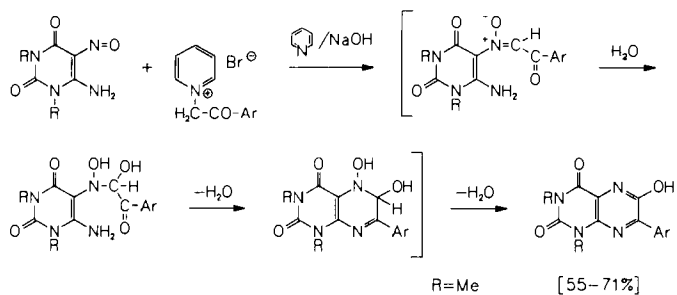
Furthermore, 6-amino-5-nitrosouracils are transformed with hydrazine hydrate and aldehydes in DMF into pyrimido[5,4-*e*][1,2,4]triazines (fervenulins) (70JHC1443; 75BCJ2884; 80JHC869). The reaction with acid hydrazides provides a versatile synthesis of pyrimido[4,5-*e*][1,2,4]triazines (isofervenuilins) (71JHC523; 78CPB367) (Scheme 66).



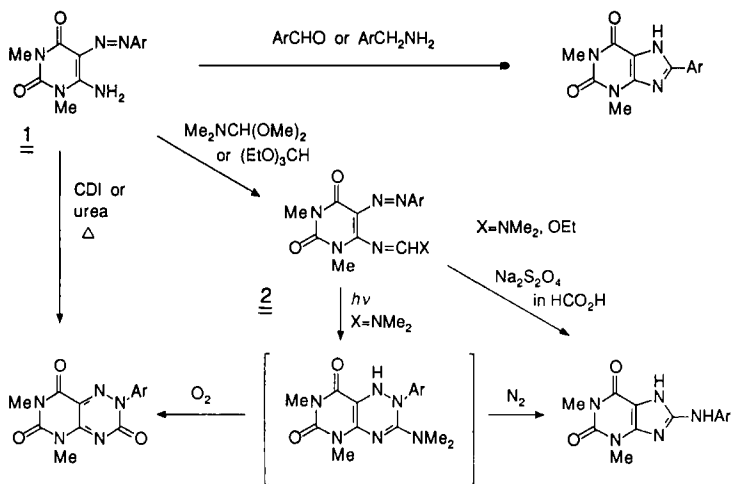
SCHEME 66

The same 6-amino-5-nitrosouracil is converted by phenacyl bromide-pyridine into a 6-hydroxy-7-phenyllumazines [77H(6)1907]. Condensation with phenacylidetriphenylphosphoranes and dimethyl acetylenedicarboxylate gives lumazine derivatives [76CC588; 81H(15)757; 82JHC949] (Scheme 67).

Fusion of 6-amino-5-arylazouracil (**1**) with benzaldehydes and benzylamines affords 8-arylxanthines [74JA5607; 77H(6)1901]. 5-Arylazo-6-methyleneaminouracil (**2**), prepared from (**1**) and DMF-DMA (or triethyl orthoformate), when reduced by sodium dithionite in formic acid, gives 8-arylaminoxanthines (77S264; 78JHC641). Pyrimido[4,5-*e*][1,2,4]triazines are accessible from **1** with *N,N'*-carbonyldiimidazole or urea (78H1387; 80JHC1365) and alternatively by photochemical cyclization of 1,2,5-hexatriene-type precursors (**2**) under aerobic conditions to give 6-aryl-1,3-dimethyl-6,7-dihydro-6-azalumazine-7-ones (77CPB2794; 80JHC1365). On the other hand, irradiation of **2** under a nitrogen atmosphere leads to the formation of 8-arylaminoxanthines via a common intermediate for the 6-azalumazines (77CPB2794) (Scheme 68).

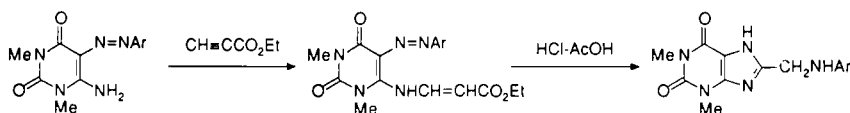


SCHEME 67



SCHEME 68

6-Amino-5-arylazouracils react with ethylpropiolate to give the Michael adducts, which are cyclized, in the presence of a mixture of hydrochloric and acetic acids, to 8-arylaminomethylxanthines [81H(16)2137; 82JHC813] (Scheme 69).



SCHEME 69

D. HALOGENO- AND HYDROXYURACILS

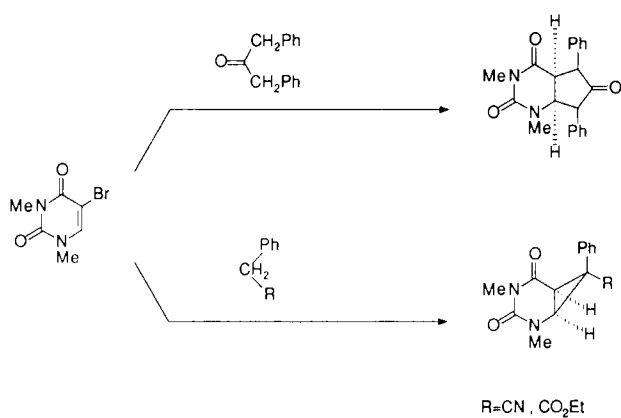
Furthermore, 5- and 6-halogeno- and hydroxyuracils have been used to synthesize several heterocondensed uracils. 5- and 6-Halogeno-1,3-dimethyluracils give, accordingly with allylamine and allylic alcohol, propargylamine and propargyl alcohols, 1,3-dialkylpyrrolo- and furo[2,3-*d*]- and [3,2-*d*]pyrimidines (72JOC2858). Similarly, 5-hydroxy-1,3-dimethyluracil and allyl bromide afford furo[3,2-*d*]pyrimidines (83H2177). After nucleophilic exchange of the halogen, Claisen rearrangement occurs, leading to 7 deazacaffeine and 7-deazatheophylline, respectively (84H2217; 85CPB4740). Analogous Claisen rearrangement of allylthiouracil derivatives leads to thieno[2,3-*d*]pyrimidines (77H(8)427] (Scheme 70).

Reaction of 5-bromo-1,3-dimethyluracil with active methylene compounds in the presence of a base causes nucleophilic addition and subsequent intramolecular substitution to give cyclopenta- and cyclopropa[*d*]pyrimidines (89CC1659). Cyclopropa[*d*]pyrimidine derivatives are also obtained by intramolecular cyclization of 6-bromomethyluracil [81JCS(P1)1896] (Scheme 71).

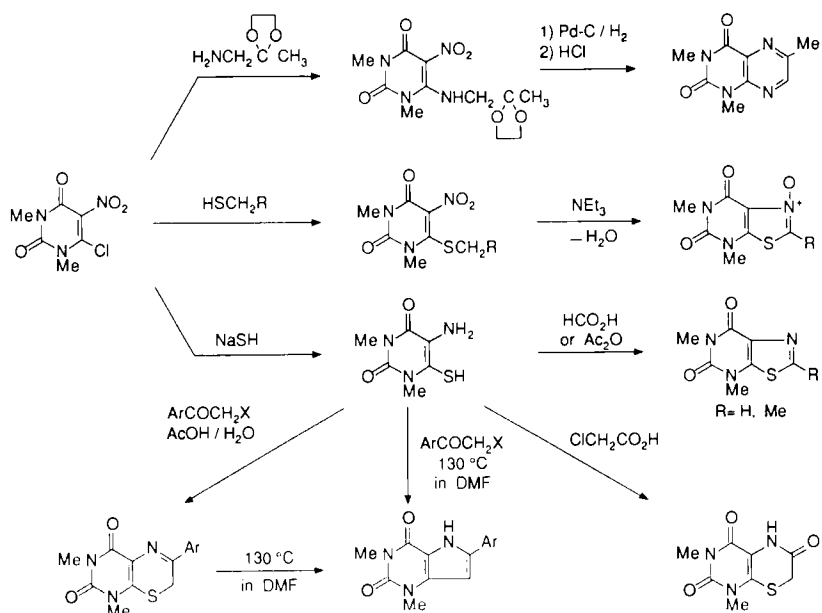
6-Chloro-1,3-dimethyluracil (64JHC212) is a convenient compound for synthesizing fused uracils. Thus, 1,3,6-trimethylumazine is prepared by reaction of 6-chloro-5-nitrouracil with 2,2-ethylenedioxypropylamine followed by catalytic reduction (67JHC124).

Thiazolo[5,4-*d*]pyrimidines are obtained accordingly, starting from 6-chloro-5-nitrouracil with mercaptans and triethylamine. The resulting *N*-oxide has proved to be an important intermediate for the preparation of several thiazolopyrimidines (81CC278; 82JHC77).

5-Amino-6-mercaptopuracil, easily prepared from 6-chloro-5-nitrouracil and sodium hydrosulfide, condenses with formic acid or acetic anhydride to furnish thiazolo[5,4-*d*]pyrimidines (89CPB2197). The condensation with chloroacetic acid and phenacyl halide gives 6-hydroxy- and 6-arylpyrim-

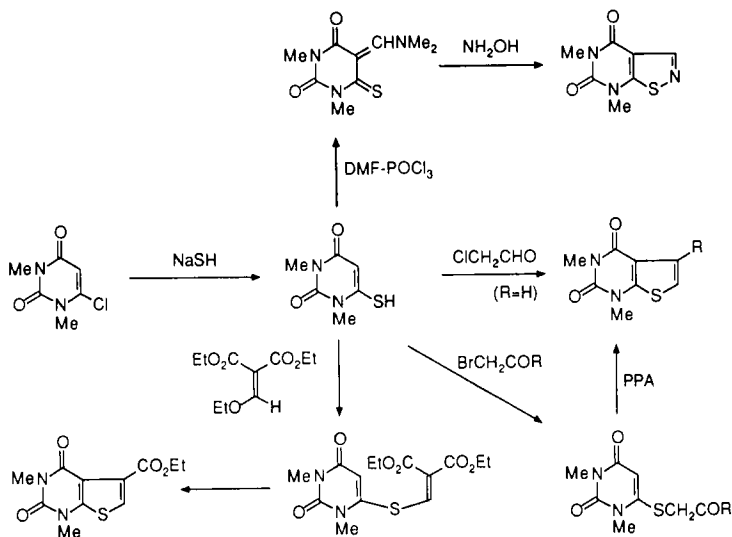


SCHEME 71



SCHEME 72

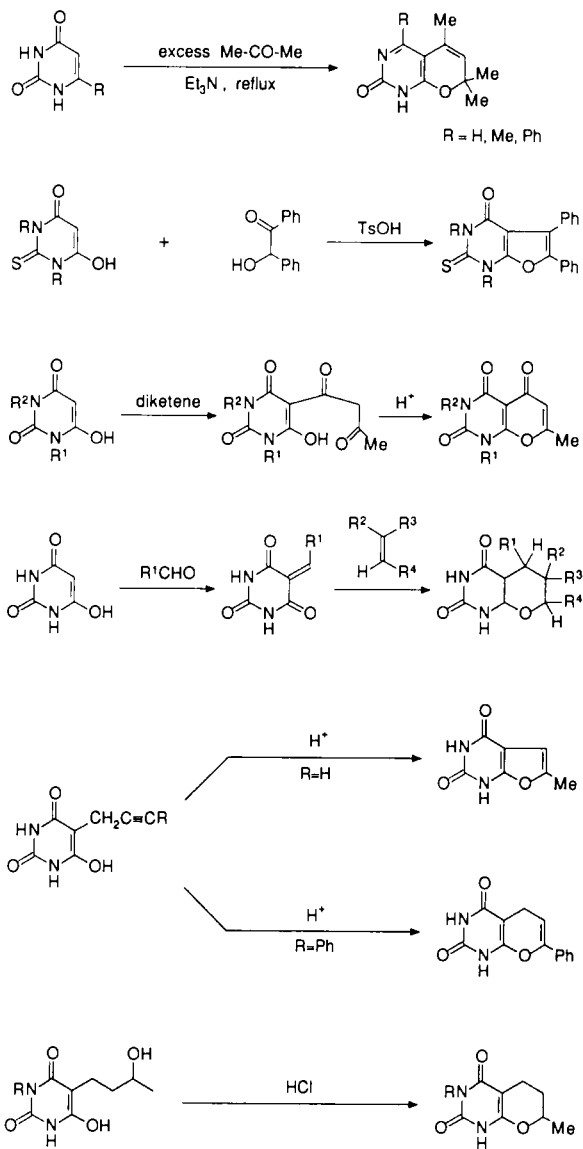
with Vilsmeier reagent (DMF-POCl₃) and hydroxylamine, with chloroacetaldehyde or α -bromoketones, and with diethyl ethoxymethylenemalonate, respectively (68CB3377; 76CPB1390; 81NKK721; 90JHC717) (Scheme 73).



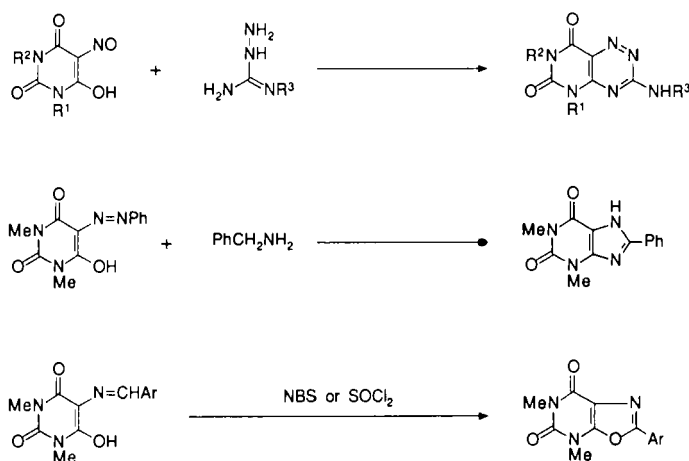
SCHEME 73

6-Hydroxyuracils (barbituric acids) are frequently used to synthesize furano- and pyrano[2,3-*d*]pyrimidines. Thus, condensation with hydrobenzoin and diketene [73CPB2639; 90IJC(B)566], cycloaddition of 5-methylenobarbituric acids with olefins (72AP354), and intramolecular cyclization between a 5-side-chain and a 6-hydroxy group (63AP235; 69YZZ266) are documented. Base-catalyzed condensation of 1-unsubstituted uracils with acetone furnishes pyrano[2,3-*d*]pyrimidines (87SC1435) (Scheme 74).

Reactions of 5-nitrosobarbituric acids with aminoguanidine, of 5-phenylazobarbituric acids with phenethylamine, and of 5-arylmethyleneaminobarbituric acid with NBS or thionyl chloride afford isofervenulins, 8-phenyl-xanthines, and oxazolo[5,4-*d*]pyrimidines, respectively [75BCJ725, 75BCJ1679; 77H(6)689, 77H(6)1901, 77H(6)1919; 78CPB765] (Scheme 75).



SCHEME 74



SCHEME 75

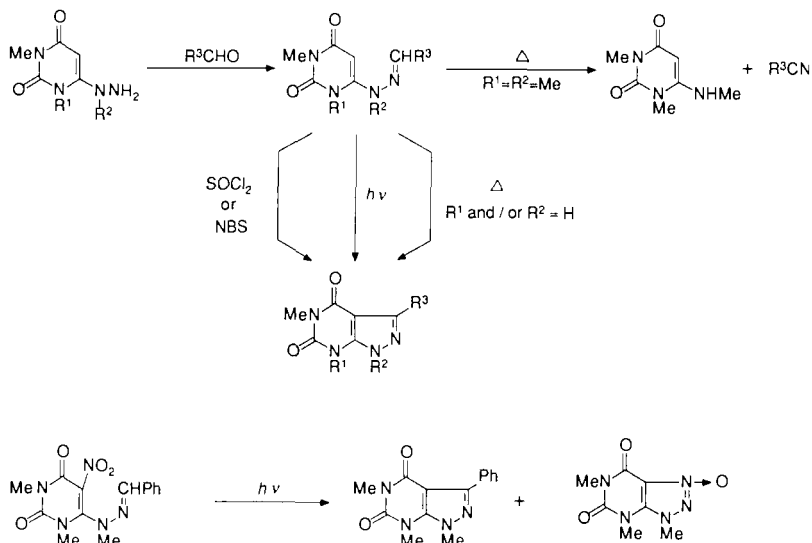
E. 6-HYDRAZINOURACILS

6-Hydrazinouracils [58LA52] are versatile starting materials for synthesizing purine analogues by adding a C-1 synthetic equivalent. They are also versatile starting materials for synthesizing fervenulins and toxoflavins by adding reactive N-units.

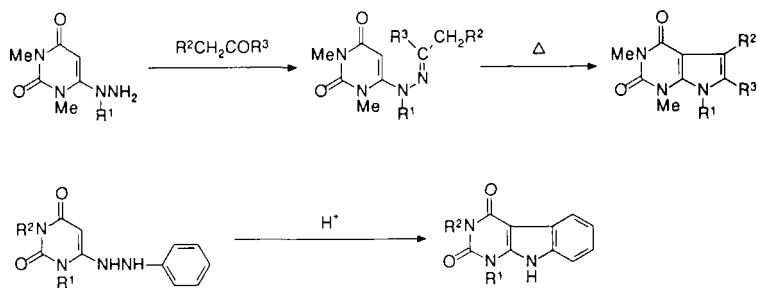
Thus, 6-benzylidenehydrazinouracils derived from 6-hydrazinouracil and aldehydes are cyclized, upon UV irradiation and treatment with thionyl chloride or NBS, to afford pyrazolo[3,4-*d*]pyrimidines. When the hydrazones are heated, cyclization to pyrazolo[3,4-*d*]pyrimidine depends on the presence of proton at the R¹ or R² positions or both; fully methylated hydrazones are decomposed into 6-methylaminouracil and nitriles [73S300; 74H153; 75BCJ1484; 77H(6)945; 84JHC969]. Photolysis of a 5-nitro derivative of the hydrazone leads to the formation of triazolo[4,5-*d*]pyrimidine *N*-oxide along with pyrazolo[3,4-*d*]pyrimidine (71CC1442; 72TL1973; 74CPB1269) (Scheme 76).

The hydrazones prepared from 6-hydrazinouracils and ketones undergo facile Fischer-type cyclization to give pyrrolo[2,3-*d*]pyrimidines (72CL367; 74CPB1459, 74CPB2921, 74JCS(P1)1921]. Heating 6-phenylhydrazinouracils under acidic conditions gives pyrimido[4,5-*b*]indoles (76JHC539; 79JHC401) (Scheme 77).

Another way to build this heterocondensed system is by reacting 6-benzylidenehydrazinouracils with NBS and acetic acid to give pyrazolo[3,4-*d*]pyrimidines (see Scheme 76) and pyrimido[4,5-*c*]pyridazines.



SCHEME 76

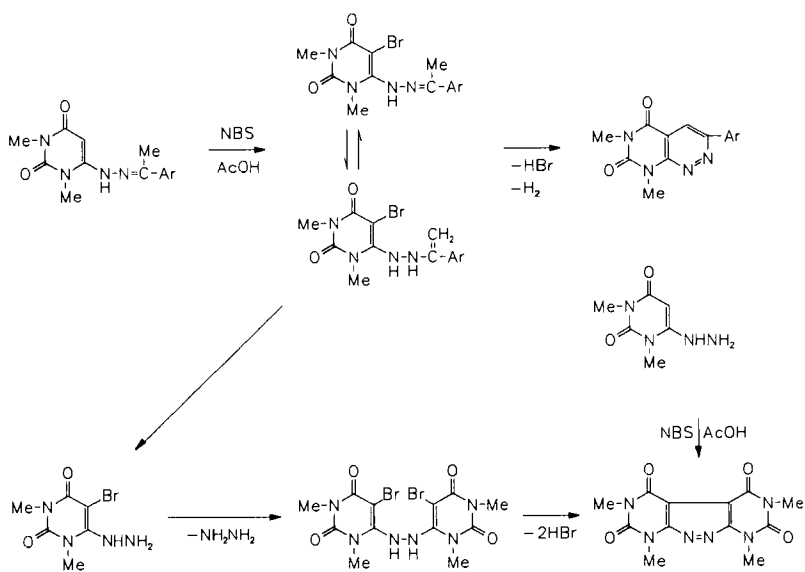


SCHEME 77

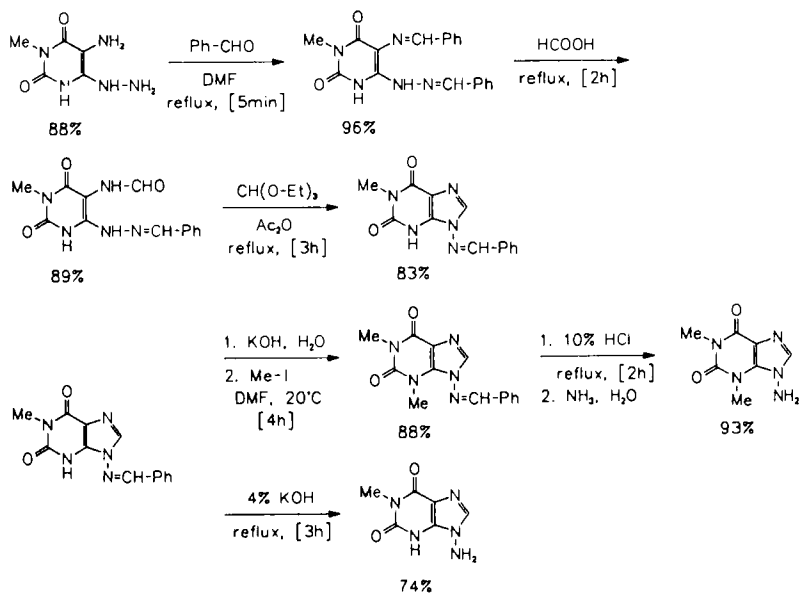
Furthermore, hydrazinouracils give, upon bromination and intermolecular hydrazine expulsion, a 2:1-adduct, which cyclizes on dehydrobromination to give a tricyclic pyridazino[3,4-*d*:6,5-*d'*]dipyrimidine (84JHC969) (Scheme 78).

A synthesis of purine derivatives 1-methyl-9-aminoxanthine and 9-amino-theophylline is based on 5-amino-6-hydrazinouracils (87KGS836) (Scheme 79).

Strongly electrophilic cyanoolefins, such as arylidenemalononitrile, arylidene-cyanoacetate, and arylidenecyanoacetamide react as C_1 reagents

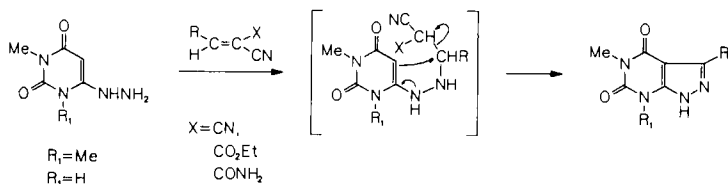


SCHEME 78



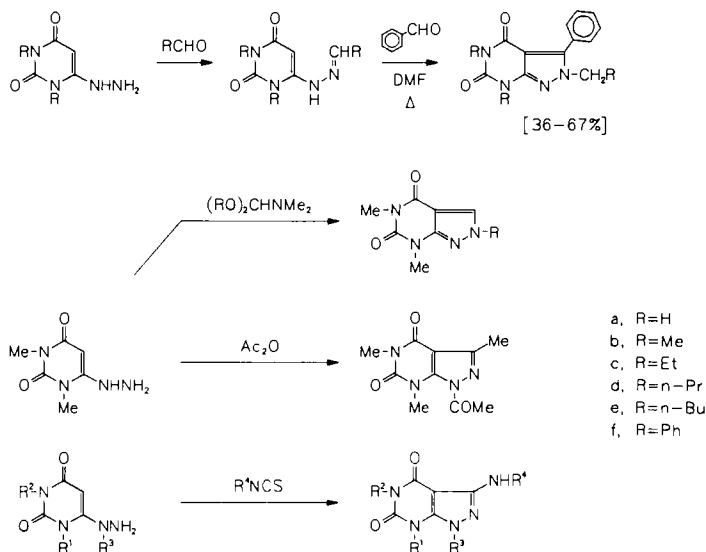
SCHEME 79

with 6-hydrazinouracils to give pyrazole[3,4-*d*]pyrimidines via Michael adducts (90JOC568). (Scheme 80).



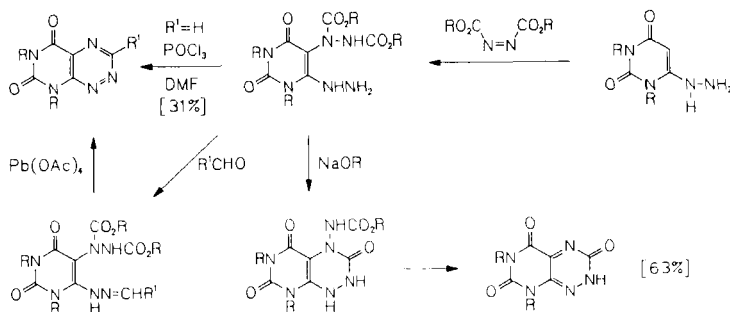
SCHEME 80

Furthermore, pyrazolo[3,4-*d*]pyrimidines are obtained by thermally induced cyclization reactions of 6-alkylidenehydrazinouracils in the presence of aldehydes [58LA42; 74JA5607; 77JCS(P1)765]. A variation is the reaction of 6-hydrazinouracils with dimethylformamide dialkylacetals and acetic anhydride accompanying *N*-alkylation and *N*-acetylation (77S176; 78JHC359). With isothiocyanates, the same pyrazolo[3,4-*d*]pyrimidine system is formed via thiosemicarbazides (79CPB1328; 80JHC1305) (Scheme 81).



SCHEME 81

Azodicarboxylates undergo Michael addition to the electron-rich 5-position. The intermediate hydrazino ester is converted with bases to fervenulones or converted with Vilsmeier reagent (POCl_3 -DMF) to fervenulins, which are available by oxidation of the corresponding hydrazone using lead tetraacetate [75JOC2321; 76JCS(P1)2398] (Scheme 82).



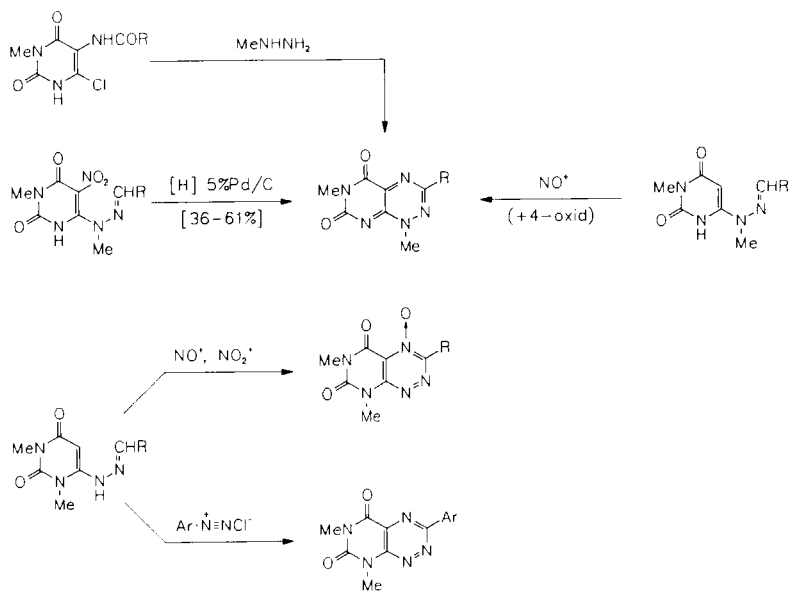
SCHEME 82

Toxoflavin was first synthesized by allowing 5-acylamino-6-chloro-3-methyluracil to react with methylhydrazine (62JA1724). Nitration and nitrosation (N1 insertion) of alkylidene- and arylidenehydrazouracils leads to toxoflavins and fervenulin 4-oxides [71CPB2647, 71TL851; 72CB3334; 73CPB448; 74JHC83; 75CPB2001, 75S177; 76JCS(P1)713; 78CPB3154; 82JHC1309; 87JHC1373]. Diazotization of arylidenehydrazinouracils affords 3-arylfervenulins via the corresponding 5-arylaazo intermediates [81H(16)559; 82JHC769] (Scheme 83).

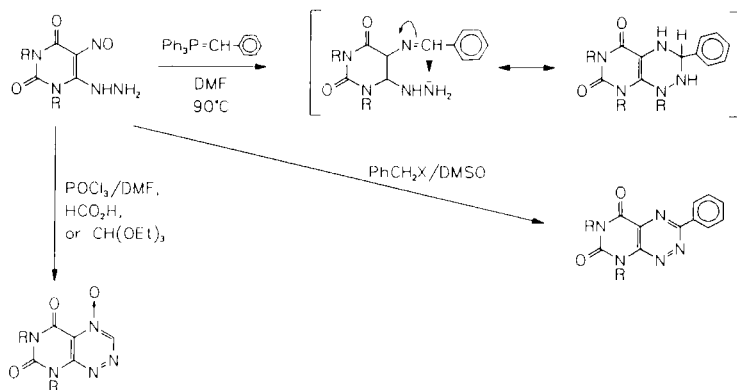
3-Arylfervenulins are formed when 6-hydrazino-1,3-dimethyl-5-nitrosouracil is allowed to react with benzylidenetriphenylphosphorane or benzyl halide-DMSO. Many of these aforementioned products are important intermediates in the synthesis of the antibiotics 2-methylfervenulin and toxaffavin [77H(6)1921; 78H29].

Normally inaccessible fervenulin 4-oxide is synthesized in a single step by reaction of 6-hydrazino-5-nitrosouracil with C_1 reagents, such as POCl_3 -DMF, formic acid and triethyl orthoformate [58LA42; 77H(6)273, 77JHC175; 78JOC469] (Scheme 84).

Treatment of 6-hydrazino-1,3-dimethyluracil with thionyl chloride leads to 4,6-dimethyl-1,2,3-thiadiazolo[4,5-*d*]pyrimidine (**1**) (76TL1129; 78JOC1677). Oxidation of 6-thiosemicarbazidouracils with *N*-chlorosuccinimide (NCS) and bromine furnishes pyrimido[4,5-*e*][1,3,4]thiadiazines (**2**) and the thiadiazolopyrimidine (**1**), respectively. The product **2** could be an intermediate for the formation of **1** because **2** is easily converted into



SCHEME 83



SCHEME 84

1 by oxidation with bromine. On heating **2** in DMF, pyrazolo[3,4-*d*]pyrimidines are formed (79CPB1965; 85JHC381).

A new route to triazolo[4,5-*d*]pyrimidines from 6-alkylidenehydrazinouracils is accomplished using *N*-nitrosodimethylamine (76CPB1917). From the starting compound, 6-aryllumazines are formed by nitrosation and subsequent reduction [77H(6)693]. Reaction of 6-(2-arylmethylene-1-methylhydrazino)uracils with sodium nitrite in acetic acid surprisingly gives oxazolo[5,4-*d*]pyrimidines. The mechanism has not been elucidated (77H(6)1925) (Scheme 85).

4-Deazatoxoflavins are accessible by reaction of 6-(1-methylhydrazino)uracils with phenacyl bromides or by intramolecular cyclization of uracil-6-ylhydrazones with a typical C-1 equivalent (triethyl orthoformate) (78H11, 78H1571, 78JHC781; 81CPB379; 82CPB172) (Scheme 86).

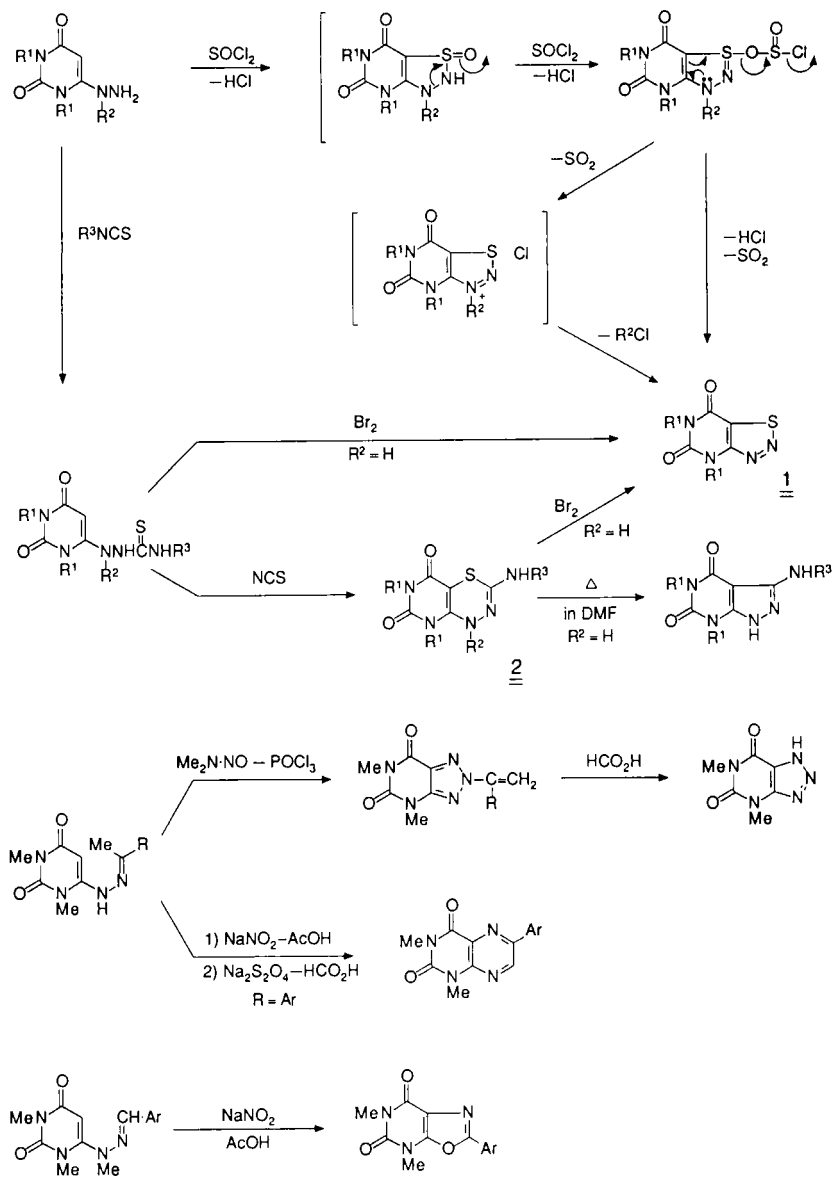
Pyrimido[4,5-*c*]pyridazines (desazatoxoflavins) are synthesized from 6-hydrazinouracils using appropriate α -diketones (58LA48; 75JHC1221; 82CL1309; 84CPB851). The condensation with aldoses gives pyrimido[4,5-*c*]pyridazine nucleosides (81CPB629). Pyrimido[4,5-*c*][1,2]diazepines are obtained by cyclization of 6-hydrazinouracil with 1,3-diketones and equivalent reagents (78S748) (Scheme 87).

6-Hydroxyuracils are good starting compounds for synthesizing isoxazolo[3,4-*d*]pyrimidines. Acid anhydrides, aromatic aldehydes, and Vilsmeier reagent are used as a C₁ reagent for forming the oxazole ring. The isoxazolopyrimidines undergo ring expansion induced by benzylamines and photochemical transposition reaction to give pyrimido[4,5-*d*]- and oxazolo[4,5-*d*]pyrimidines, respectively (77CPB2974; 78CPB2497; 79CL155; 81NKK721; 84JHC267) (Scheme 88).

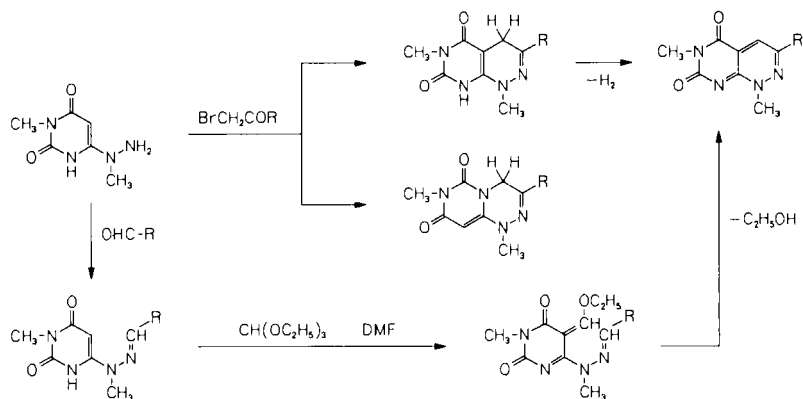
F. AZIDOURACILS

6-Azido-5-formyl-1,3-dimethyluracil is cyclized with hydrazines to afford pyrazolo[3,4-*d*]pyrimidines. The irradiation in methanol (or heating in tetralin) and the reaction with triphenylphosphine in benzene result in the formation of isoxazolo[3,4-*d*]pyrimidine and pyrimido[4,5-*d*][1,2,3]triazine, respectively (83CPB3959). Furazano[3,4-*d*][1,2,3]pyrimidine 1-oxide (cf. Scheme 65) is synthesized by heating 6-azido-5-nitrouracil in DMF (73JHC415, 73JHC993) (Scheme 89).

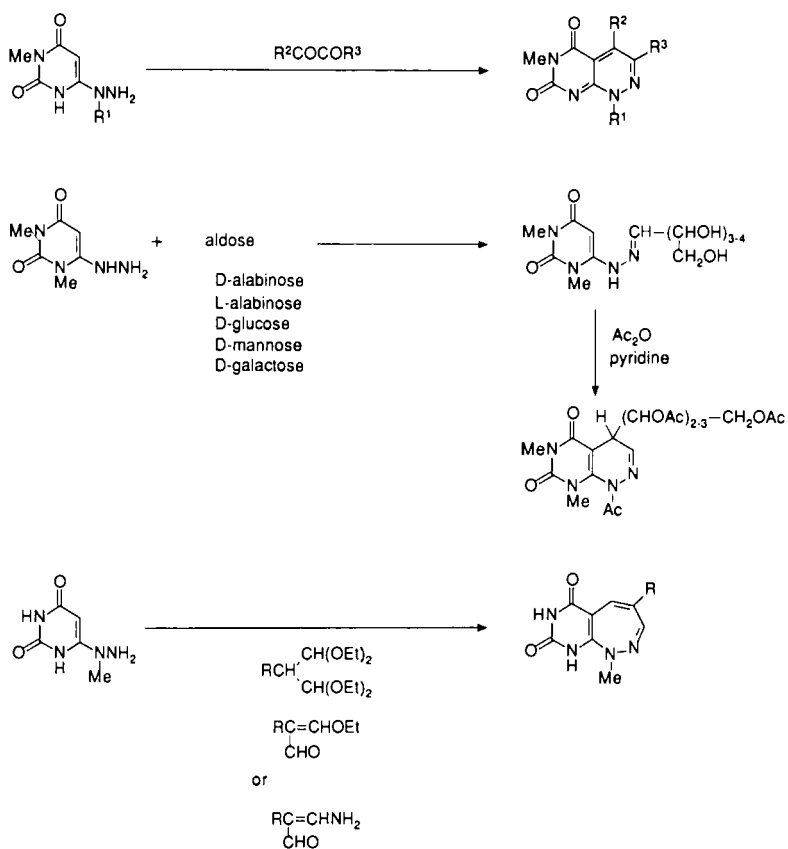
6-Azido-1,3-dimethyluracil is converted into 1,2,3-triazolo[4,5-*d*]pyrimidine (R = H) in a reaction with potassium carbonate in DMF, and the presence of alkyl halides affords 1-alkylated products [77H(6)1915]. Another study reports that the reaction with methyl iodide gives two



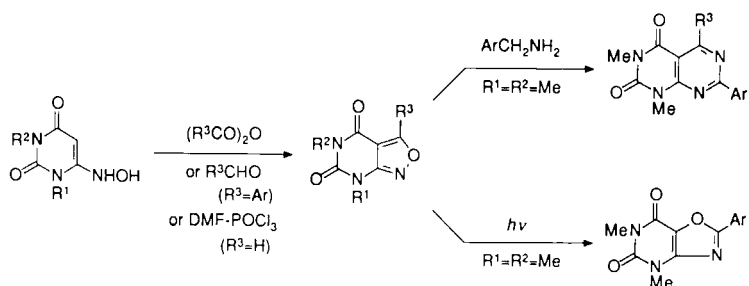
SCHEME 85



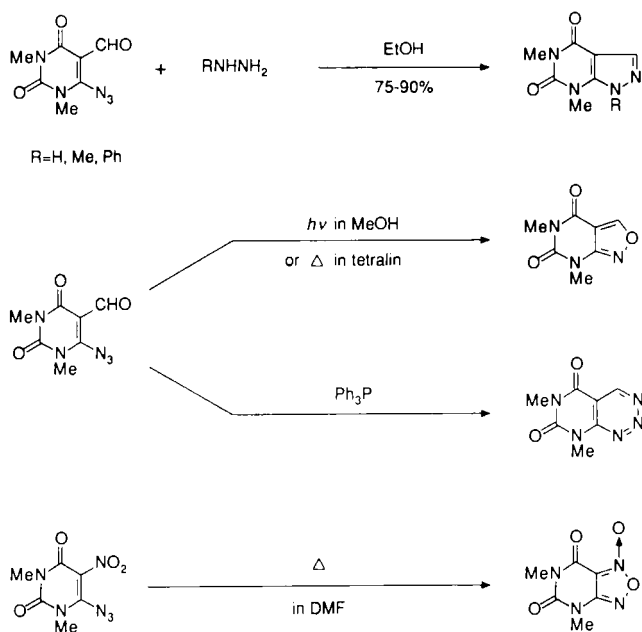
SCHEME 86



SCHEME 87

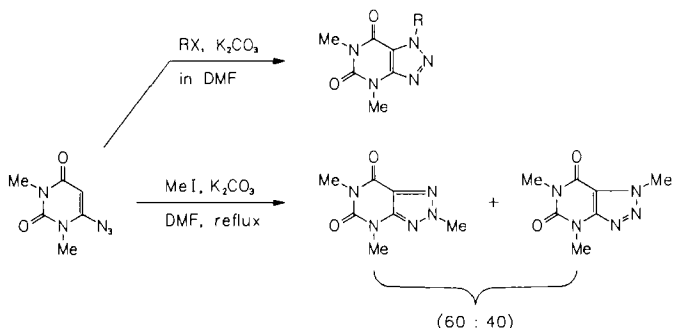


SCHEME 88



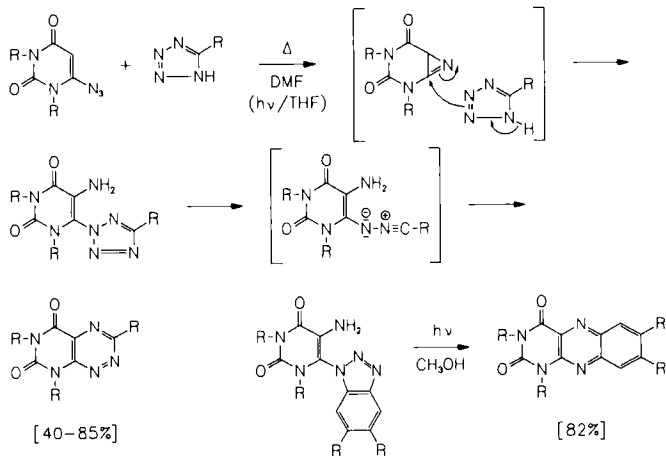
SCHEME 89

isomeric 1- and 2-methyltriazolopyrimidines under similar conditions (87BSB659) (Scheme 90).



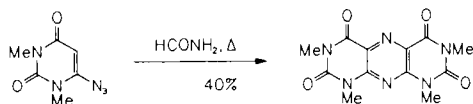
SCHEME 90

6-Azidouracil forms fervenulins on heating with tetrazoles via a complex reaction mechanism, while UV irradiation of 6-azidouracils in the presence of benzotriazoles gives alloxazines [81H(15)285] (Scheme 91).



SCHEME 91

Two molecules of 6-azido-1,3-dimethyluracil lead to pyrimido[5,4-*g*]pteridine-2,4,5,7-tetrone in a thermal reaction (in formamide) (82CPB3377), although the photochemical reaction gives pyrimido[4,5-*g*]pteridine-2,4,6,8-tetrone (see Scheme 37) (Scheme 92).



SCHEME 92

G. 6-METHYLURACILS

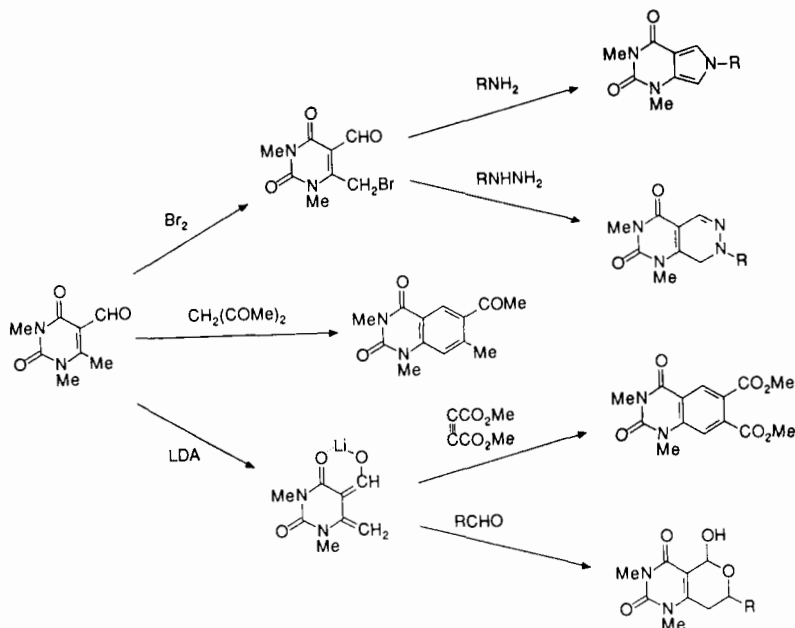
6-Methyluracil derivatives possessing a functional group at C-5 have proved to be favorable to heterocyclic annulation. In particular, an electron-withdrawing group, such as nitro or formyl, activates the 6-methyl group.

Thus, starting from 1,3,6-trimethyl-5-nitrouracil (**1**), pyrrolo[3,2-*d*]pyrimidines (9-deazaxanthines) can be synthesized three ways using DMF-DMA, benzaldehydes, and benzyl halides. Condensation of the 6-methyl group with DMF-DMA and subsequent reduction of the 5-nitro group result in ring formation to give pyrrolo[3,2-*d*]pyrimidines (74CPB2593; 88KFZ185). When the 6-methyluracil (**1**) and benzaldehydes are heated, the product depends on the solvent used. The reaction in DMF and ethanol gives 7-hydroxypyrrolopyrimidines (**2**) and 6-styryluracils, respectively (80CPB1636; 81CL1273; 82CPB3187). The latter product is cyclized to pyrrolo[3,2-*d*]pyrimidine (**3**) on reduction or treatment with triethylphosphite, and irradiations in benzene and isopropanol furnish **2** and **3**, respectively (65JOC655; 77CPB563). The stable sodium salt of **1** is isolated upon treatment with sodium ethoxide; it reacts with benzyl halides in the presence of potassium carbonate to afford 5-hydroxypyrrolo[3,2-*d*]pyrimidines via benzylation at the 6-methyl group [82S1097; 84JCS(P1)583] (Scheme 93).

Pyrrolo[4,3-*d*]pyrimidine 1-oxides, though difficult to obtain, are available by a one-step synthesis from 6-bromomethyl-1,3-dimethyl-5-nitrouracil. Condensation at 0°C allows isolation of the alkylamino intermediates, which, in boiling ethanol, cyclize to the 1-oxides. The *N*-oxides, substituted with a benzyl group at N-2, undergo ring expansion in the presence of sodium ethoxide to give pyrimido[5,4-*d*]pyrimidines [77CC556; 82JCS(P1)277]. The alkylamino intermediates are also used to synthesize pyrimido[5,4-*d*]pyrimidines by reduction and subsequent treatment with triethyl orthoformate (81CPB3060) (Scheme 94).

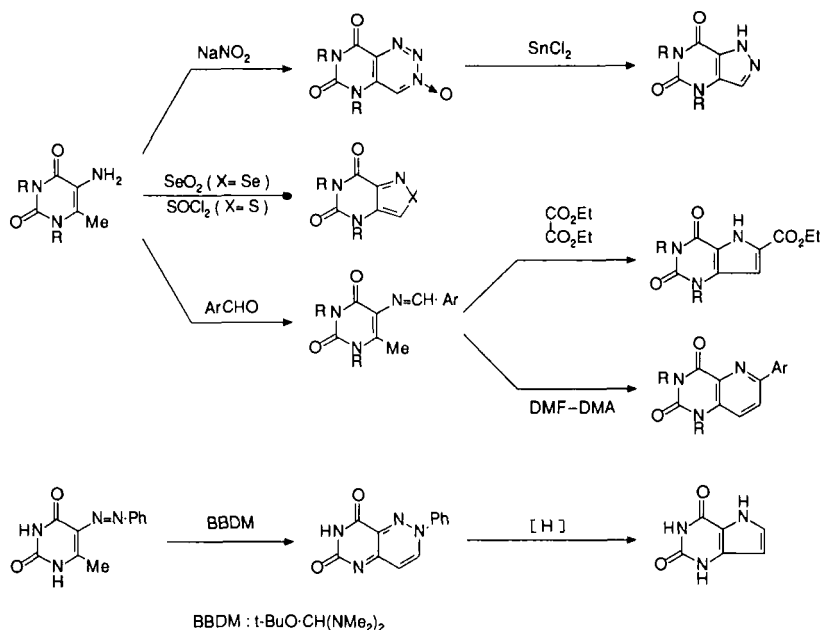
A novel and simple approach to pyrrolo[3,4-*d*]pyrimidines and pyrimido[4,5-*d*]pyridazines is bromination of 5-formyl-1,3,6-trimethyluracil and subsequent cyclization with amines and hydrazines, respectively (78S463;

81CPB1525). The 5-formyluracil is converted into quinazolines by condensation with acetylacetone (81JOC3949) or by cycloaddition of the lithium dienolate to olefins (80TL531). Aldehyde dienophiles lead to pyrano[4,3-*d*]pyrimidines [81H(15)289]. (Scheme 95).



SCHEME 95

5-Amino-6-methyluracil derivatives are used to synthesize various types of condensed pyrimidines. Thus, the reaction with sodium nitrite, selenium dioxide, and thionyl chloride leads to pyrimido[4,5-*d*][1,2,3]triazine 3-oxides (63JOC1329; 65JOC199; 70JHC405), isoselenazolo[4,3-*d*]pyrimidines, and isothiazolo[4,3-*d*]pyrimidines, respectively (85S695; 87TL4579). 6-Arylideneamino-6-methyluracil, obtained from 5-aminouracil and benzaldehydes, condenses with diethyl oxalate and DMF-DMA to cyclize to pyrrolo[3,2-*d*]pyrimidines (57CB738) and pyrido[3,2-*d*]pyrimidines, respectively (80S479; 82JHC805). Pyrimido[5,4-*c*]pyridazines, formed by condensation of 6-methyl-5-phenylazouracil with *t*-butoxybis(dimethylamino)methane (BBDM), undergo reduction to a simple pyrrolo[3,2-*d*]pyrimidine (9-deazaxanthine) (78JOC2536) (Scheme 96).



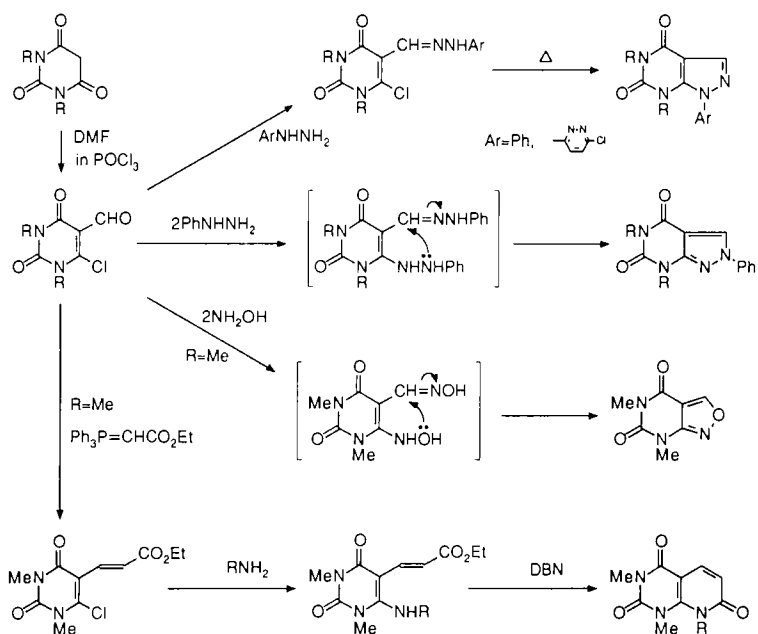
SCHEME 96

H. 6-CHLORO-5-FORMYLURACILS

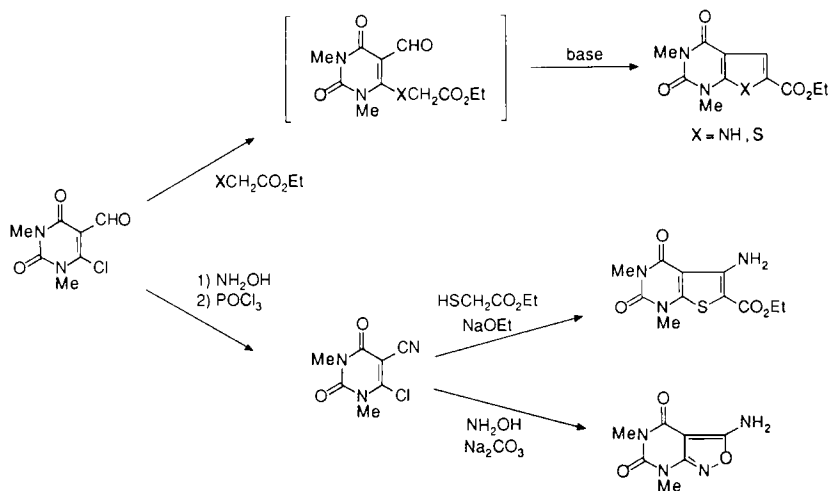
6-Chloro-5-formyl-1,3-dimethyluracil is a starting material that is easily accessible by Vilsmeier formylation of the commercially available 1,3-dimethylbarbituric acid (71YZ1372).

6-Chloro-5-formyluracils react, on heating with an equivalent of arylhydrazines via appropriate hydrazone formation, to form 1-substituted pyrazolo[3,4-*d*]pyrimidines. Using two equivalents of phenylhydrazine, however, affords 2-substituted pyrazolo[3,4-*d*]pyrimidines (72CPB399; 84H513). Similar treatment with excess hydroxylamine leads to the formation of isoxazolo[3,4-*d*]pyrimidines (81NKK721). The Wittig olefination, after exchange of a 6-chloro atom for an amino group, gives pyrido[2,3-*d*]pyrimidines (84H513; 85H2057) (Scheme 97).

Furthermore, 6-chloro-5-formyluracil, in reaction with ethyl glycinate and ethyl thioglycolate, furnishes pyrrolo- and thieno[2,3-*d*]pyrimidines, respectively. Amino-substituted thieno[2,3-*d*]pyrimidines and isoxazolo[3,4-*d*]pyrimidines are synthesized starting from 6-chloro-5-cyano-1,3-dimethyluracil (74CPB2921; 78CPB3208; 81NKK721; 90JHC717) (Scheme 98).



SCHEME 97



SCHEME 98

5-Formyl-6-chlorouracils are compounds that are well designed for heterocyclization reactions. With acylated hydrazines and ethylenediamines, pyrazolo[3,4-*d*]pyrimidines and pyrimido[3,4-*e*][1,4]diazepines are formed (85UP1). Wittig reaction affords the 6-chloro-5-ethenylcarboxylic esters which give with hydrazines more pyrazolo[3,4-*d*]pyrimidines (84H513; 85CZ188). Ethoxycarbonyl-methylenetriphenyl-phosphorane reacts with 6-chloro-5-formyl-1,3-dimethyluracil in an unexpected formation of a novel uracil phosphorane [91PS(ip)] (Scheme 99).

5-Deazaflavines and pyrido[2,3-*d*;6,5-*d'*]dipyrimidines, which possess an ability to oxidize an alcohol, are obtained from 6-chloro-5-formyl-3-methyluracil and anilines or 6-aminopyrimidines [76CC203; 78CPB3208; 81JA5943; 84TL(25)1741; 86JHC241]. This method is applied to the total synthesis of coenzyme factor 420 [90JCS(P1)253] (Scheme 100).

Benzothiopyrano[2,3-*d*]pyrimidines (5-deaza-10-thiaflavines) are of special biological interest because of their isosteric and isoelectronic structure to 5-desazaflavine. The synthesis starts from 6-chloro-5-formyl-3-methyluracil and thiophenol and subsequent cyclization of the phenylthio intermediate with PPA. Base-catalyzed reduction with secondary alcohols leads to a 5*H*-benzothiopyrano[2,3-*d*]pyrimidine (78TL2803; 81JHC1329). 8-Substituted 5-deazaflavins have been made by a simple approach (cf. (85JHC841) (Scheme 101).

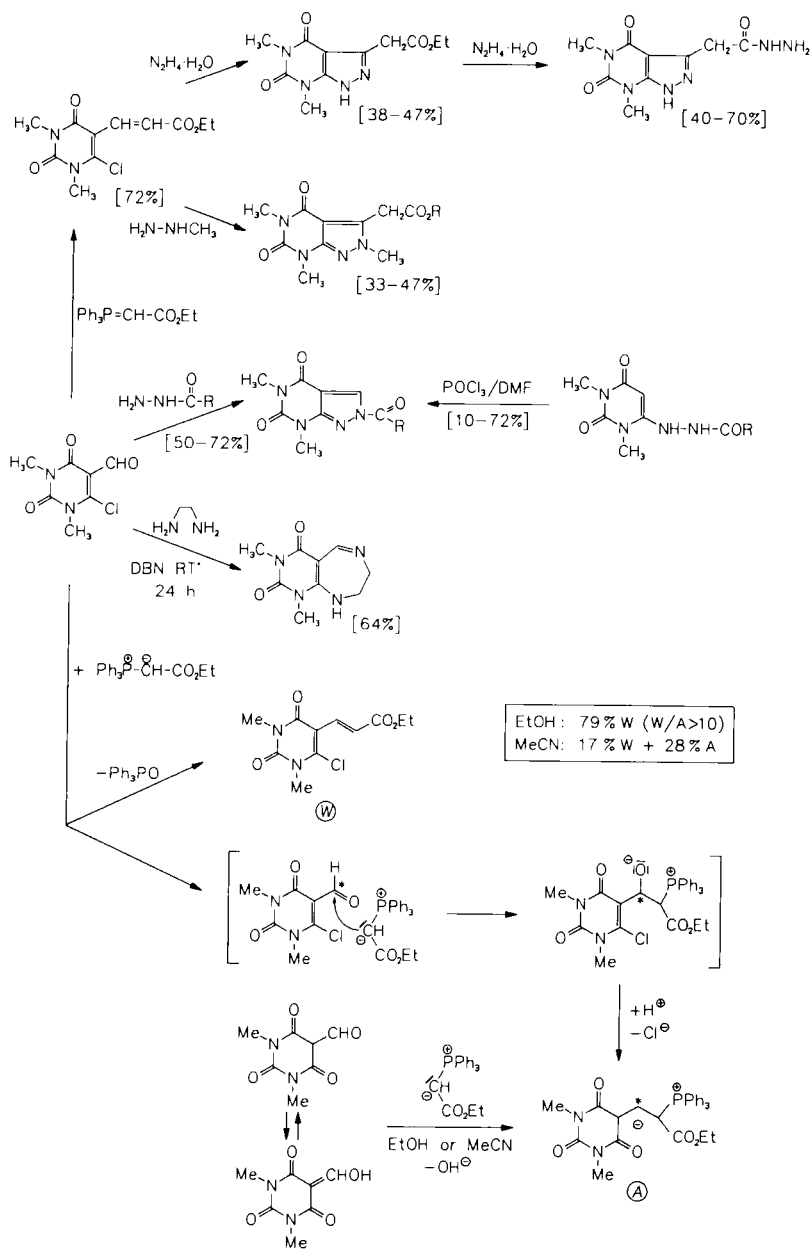
2-Amino- and 2-benzylpyridine, upon heating with 6-chloro-5-formyl-3-methyluracil in DMF afford tri- and tetraazaphenanthrenediones (so called bent-5-desazaflavines) (80CL817) (Scheme 102).

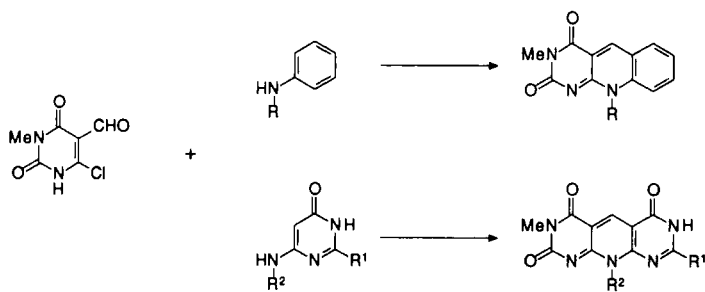
Recently, the 6-chloro atom has been nucleophilically exchanged by 2-arylamino-methylfuran and -thiophene. The formyl group then transformed into an oxime and nitriloxide function. Via intramolecular 1,5-dipolar cycloaddition, a complex spiro-linked pyrido[2,3-*d*]pyrimidine has been obtained [86CPB3994; 88JCS(P1)607, 88S342] (Scheme 103).

6-Chloro-5-formyl-1,3-dimethyluracil reacts, furthermore, with 2-aminothiophenole in the presence of 1,5-diazabicyclo-[4,3,0]non-5-ene (DBN) via a Smiles rearrangement to afford pyrimido[4,5-*b*][1,4]benzothiazine, while, without base, a simple condensation-substitution step leads to the formation of pyrimido[4,5-*b*]-[1,5]benzothiazepines (85UP1) (Scheme 104).

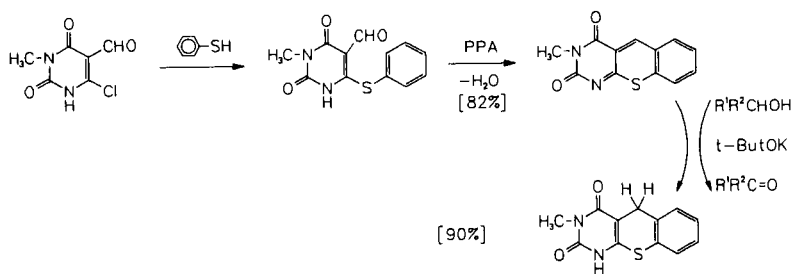
I. 5-BENZOYLURACILS

While 5-formyluracils are easily accessible by a Vilsmeier formylation reaction, the first synthesis of 5-benzoyluracil could be achieved only by a multistep procedure involving lithiated pyrimidines (56JA2136; 63JMC550).

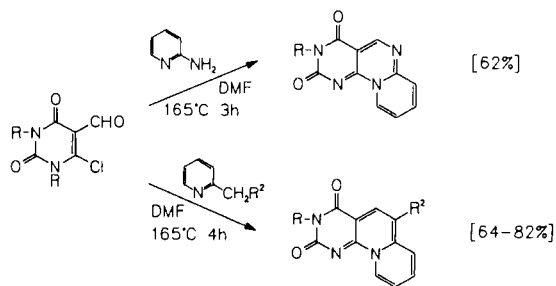
SCHEME 99. *, ^{13}C -labelled.



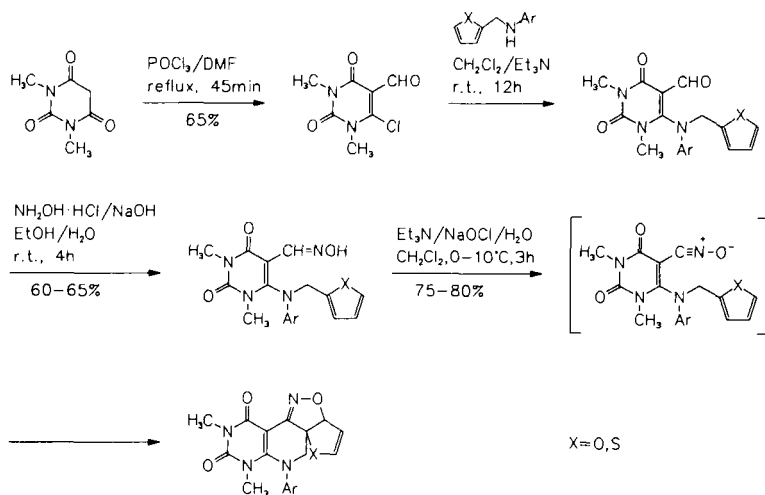
SCHEME 100



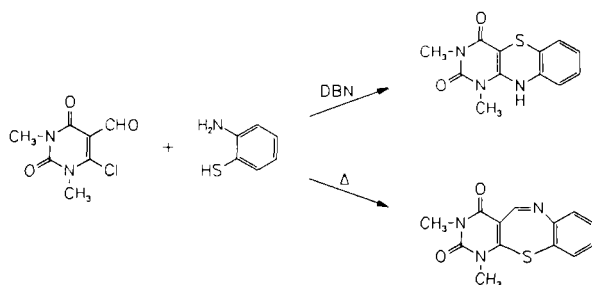
SCHEME 101



SCHEME 102



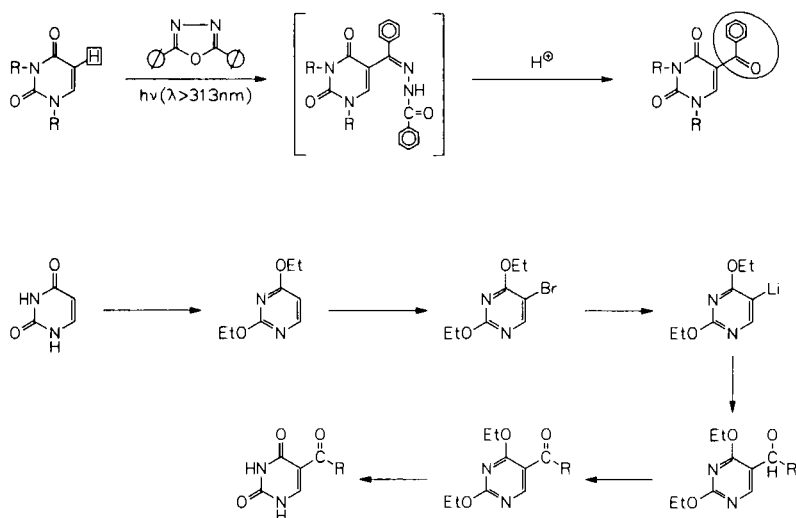
SCHEME 103



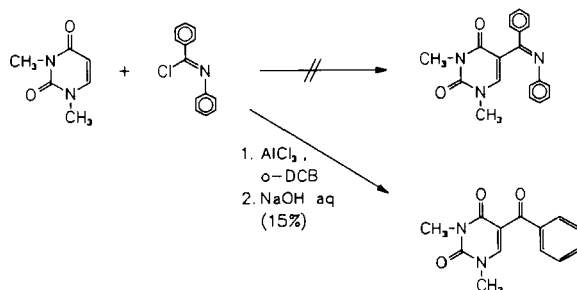
SCHEME 104

An elegant and photoselective 5-benzoylation was found upon photoreaction of 1,3-dimethyluracil with 2,5-diaryl-1,3,4-oxadiazoles (68TL3971; 73H1101, 73T41; 76CL153; 77BCJ3281, 77JOC1496). Instead of the expected [2 + 2]-photocycloaddition, a ring-cleaving addition of the electron-poor C-2 occurs at position 5 of the uracil. Saponification leads to 5-benzoyl-1,3-dimethyluracil [80CB(113)2566] (Scheme 105).

While direct benzoylation under Friedel–Crafts conditions is not successful, it has been found that diphenylimidoylechloride reacts under AlCl_3 catalysis in a one-pot reaction to give 5-benzoyluracil [92MI1(up)] (Scheme 106).



SCHEME 105



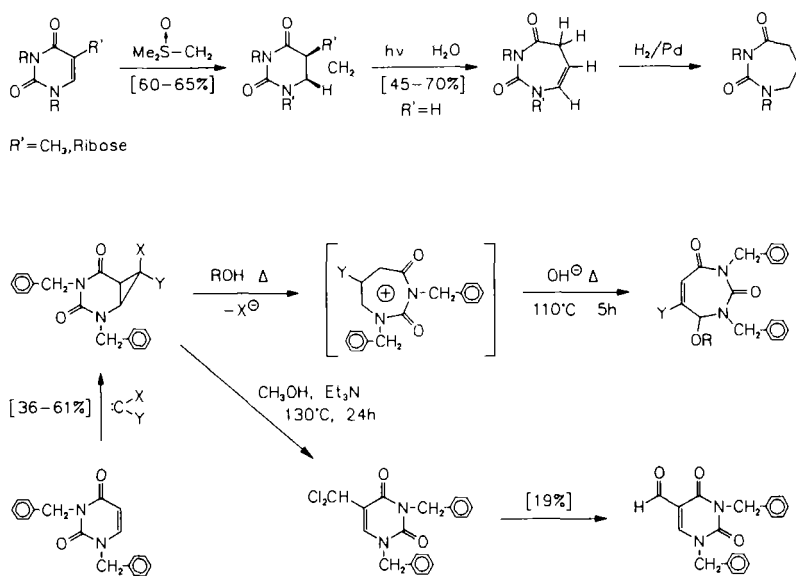
SCHEME 106

VIII. Cycloaddition Reactions to the 5,6-Double Bond; Ring Enlargement Reactions

As shown previously, the 5,6-double bond of uracil and 1,3-dimethyluracil shows a considerable polarization (cf. Sec. II). Thus, their readiness towards cycloaddition reactions has been investigated. However, the cyclophilic reactivity of uracils is rather low, especially in view of thermal cycloadditions, such as Diels–Alder and 1,3-dipolar cycloadditions.

Carbenes have been found to afford [2 + 1]-cycloadducts in good yield. The resulting 5,6-methylenepyridines (2,4-diazabicyclo[4.1.0]heptan-3,5-

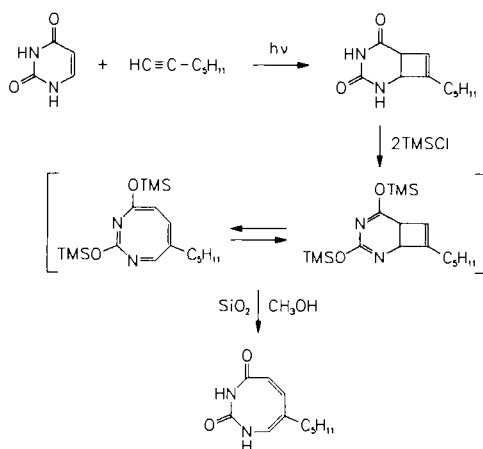
diones) are converted, upon UV irradiation, to give seven-membered 1,3-diazepinediones (69JA7751; 71JA3478). Similarly, dihalocarbene adds to the 5,6-double bond; the intermediate bicyclic compound rearranges by heating in methanol to afford 1,3-diazepinediones; however, applying forcing conditions, the annellated cyclopropane ring is cleaved to give, again, the more stable uracil moiety [74H467; 75H707; 76H(5)19; 77H(8)609, 77T1493, 77T2603, 77T2609] (Scheme 107).



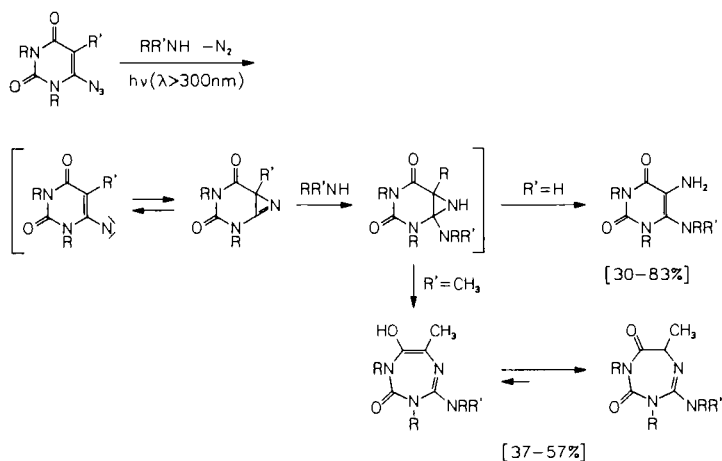
SCHEME 107

Interestingly, the 5,6-double bond of uracil takes part in a cycloaddition reaction when it is incorporated into diene and azadiene chromophors (see Scheme 163) (88TL4401; 89CB1673). However, photoinduced [2 + 2]-cycloaddition of heptyne to uracil, with subsequent *O*-silylation and electrocyclic ring enlargement, leads to a 1,3-diazocine, which gives, after saponification, a stable eight-membered 1,3-diazocine-(1*H*,3*H*)-2,4-dione (83JOC2337) (Scheme 108).

An internal cycloaddition is known to start from 6-azido-1,3-dimethyluracil. Upon UV irradiation, denitrogenation occurs to form an intermediate nitrene, which cyclizes to give a bicyclic azirine, and is then intercepted by amine addition. When the 5-substituent is a methyl group, ring enlargement takes place to afford a 1,3,5-triazepine [78TL1531; 84JCS(P1)1719]; for R = H, the aziridine ring is cleaved to give 5,6-diamino-1,3-dimethyluracil (76CC731) (Scheme 109).



SCHEME 108



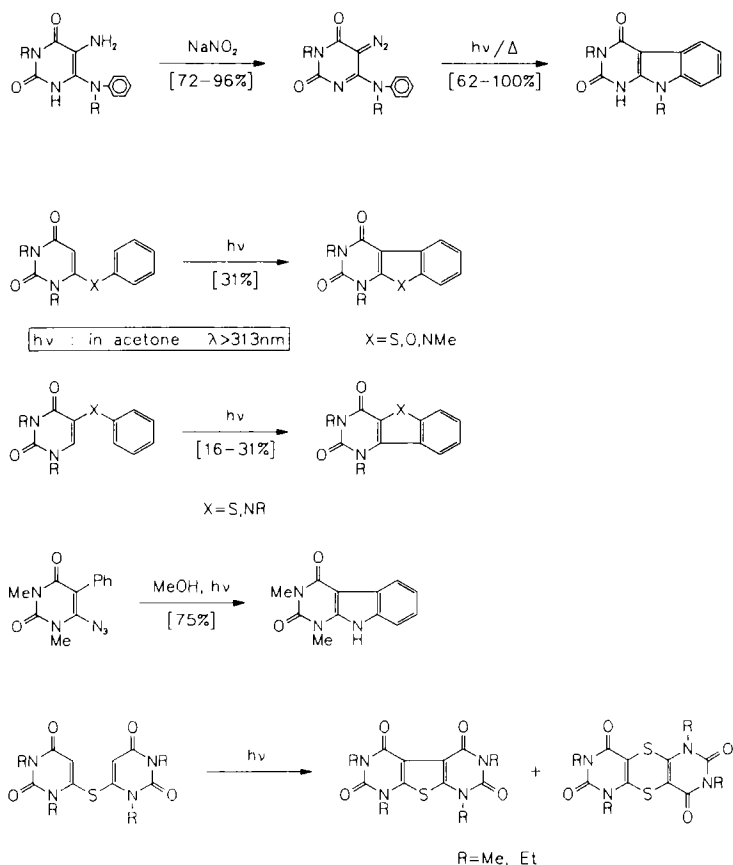
SCHEME 109

IX. Bi-, Tri-, and Oligocyclic Systems Resulting from Uracils

In addition to these aforementioned fundamental reactions on the intact uracil molecule, suitable substituted uracil derivatives have proven to be versatile starting materials of typical synthon character, leading to novel condensed heterocyclic systems consisting of two, three, and more heterocyclic rings. Many of these possess great biological interest [antibiotics,

natural compounds, analogues of the reduced nicotinamide adenine dinucleotide phosphate (NADPH) model] [80MI3; 88JCS(P1)1809, 88JHC549; 89CC44].

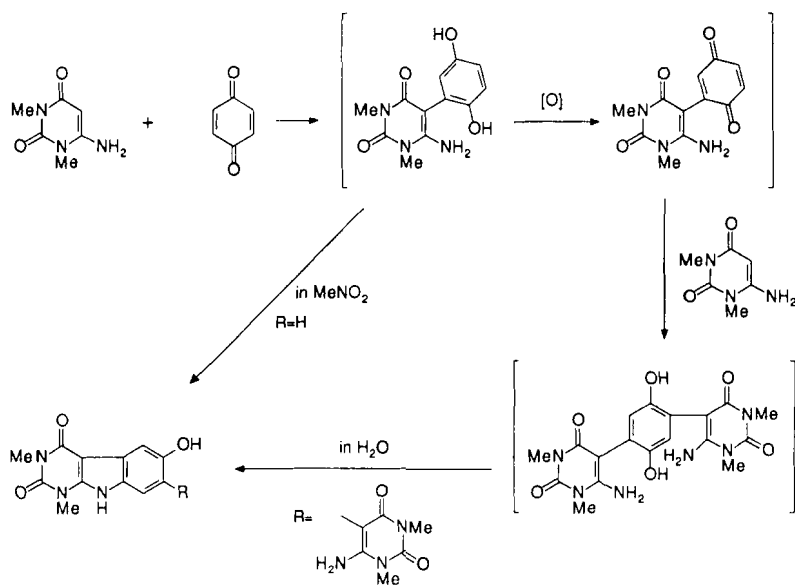
6-Anilino-5-diazouracils, prepared by diazotation, are thermally or photochemically (via a carbene intermediate) transformed into pyrimido[4,5-*b*]indoles [77H(6)1911]. Furthermore, 6-phenylthio-6-(*N*-methylanilino)- and 6-anilino-1,3-di-methyluracil can be photochemically cyclized into benzothieno[2,3-*d*]pyrimidine and pyrimido[4,5-*b*]indole [75JCS(P1)503; 83CPB3959]. Photocyclization of bis-6,6'-uracilyl sulfides unexpectedly leads to the formation of thieno[2,3-*d*:4,5-*d'*]dipyrimidines along with [1,4]dithiino[2,3-*d*:5,6-*d'*]dipyrimidines (77TL2595; 81CPB1039) (Scheme 110).



SCHEME 110

Furthermore, 6-chloro-1,3-dimethyl-5-vinyluracils are readily transformed into the 6-azido derivatives. UV irradiation in acetone leads, under denitrogenation, to nitrenes, which insert into the sidechain to give pyrrolo[2,3-*d*]pyrimidines in high yield. Analogously, 6-chloro-5-styryluracils are smoothly photoconverted in acetone solvent to afford, under 6π -electron cyclization, benzo[*h*]quinazoline-diones (85UP1).

One-step synthesis of 6-hydroxypyrimido[4,5-*b*]indole involves the condensation of 1,3-dimethyl-6-aminouracil with *p*-benzoquinone (66CB3524; 81JOC4197) (Scheme 111).

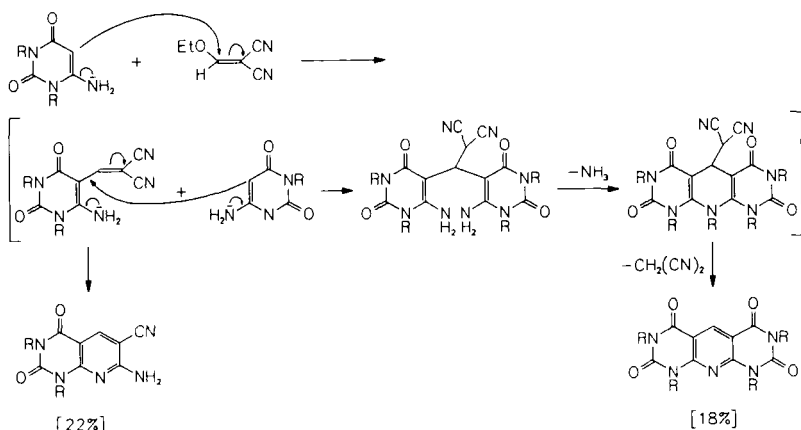


SCHEME 111

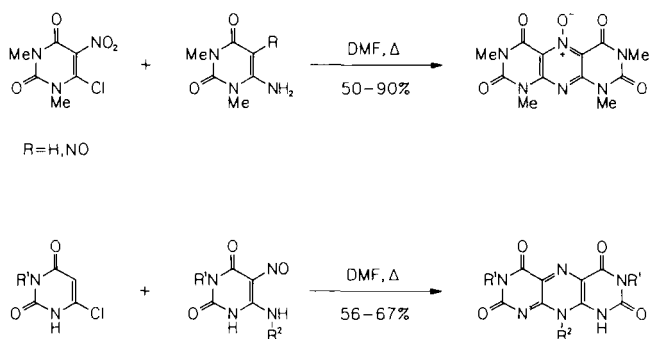
6-Amino-1,3-dimethyluracil reacts with ethoxymethylene-malonodinitrile in a complex reaction pathway to give 1,3,7,9-tetramethylpyridol[2,3-*d*:6,5-*d*]dipyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetraone as well as a 7-aminopyrido[2,3-*d*]pyrimidine-6-carbonitrile (78H197) (cf. Scheme 48) (Scheme 112).

6-Chloro-5-nitouracil and 6-aminouracils give, upon heating in DMF, a high yield of pyrimido[5,4-*g*]pteridine 5-oxide (71TL4271). 10-Substituted pyrimido[5,4-*g*]pteridines, prepared by condensation of 6-alkylamino-5-nitrosouracils with appropriate 6-chlorouracils, oxidize cyclopentanol to give cyclopentanone (72CPB2063; 82CL1127) (Scheme 113).

6-Amino-3-methyluracil reacts with arylaldehydes in a 2 : 1 ratio to give



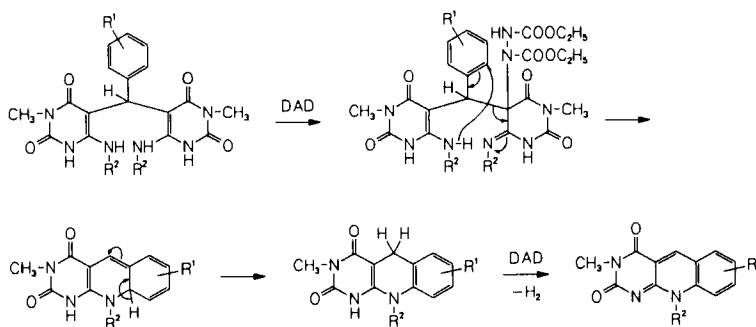
SCHEME 112



SCHEME 113

arylbis-(6-amino-3-methyluracil-5-yl)methane. Melting with azodicarboxylates (DAD) at 160°C gives, in a complex mechanism, tri- and tetracyclic pyrido-pyrimidines, such as 1,3-dimethylpyrimido[4,5-*b*]quinoline-2,4(1*H*,3*H*)-diones (5-deazaflavins) [78CC764; 79CPB2507; 80CPB3049, 80JCS(P1)978; 85JHC841, 85JHC873]. *o*-Halogeno-benzaldehydes provide a one-step synthesis of 5-deazaflavins [82CC1085; 84JCS(P1)561]. Acid hydrolysis of the bispyrimidinyl-(5)-methane derivatives lead to octahydrodipyrimido[4,5-*b*:5',4'-*e*]pyridines and octahydro-dipyrimido[4,5-*b*:5',4'-*e*]pyrans (66CB3530) (Scheme 114).

Tricyclic-fused pyrimidines containing sulfur atoms are accessible from various uracil derivatives. 10-Thiaisoalloxazines are synthesized starting from 6-(*o*-amino-phenylthio)uracils after diazotization, azide exchange,

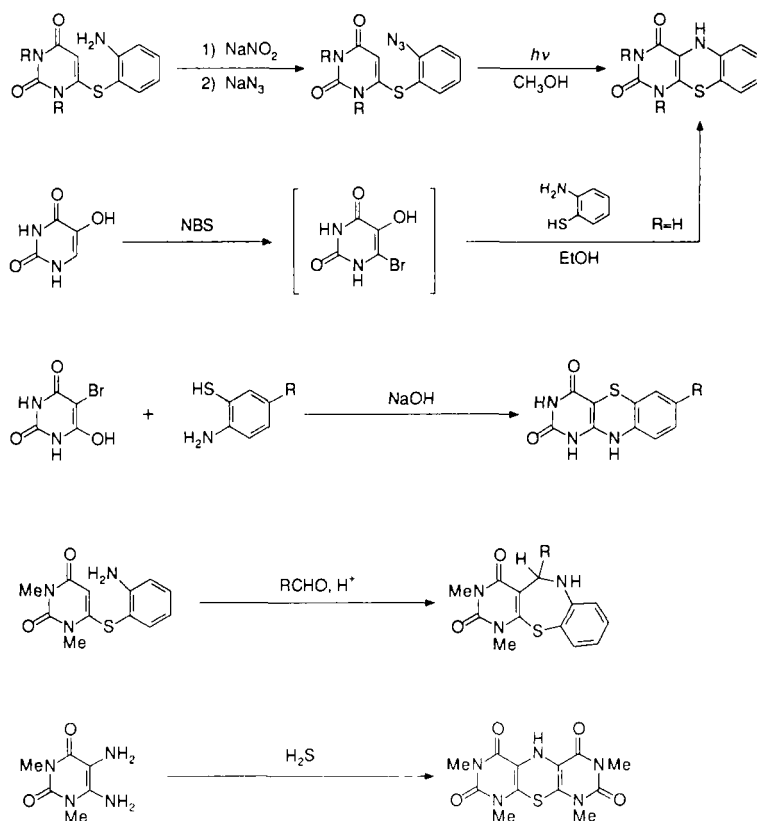


SCHEME 114

and finally photo-induced nitrene formation (77CC557). An alternative approach consists of bromination of 5-hydroxyuracil and subsequent condensation with *o*-aminothiophenol. This method is applicable to the synthesis of pyrimido[4,5-*b*][1,4]thiazines (84CPB2474). On the other hand, pyrimido[5,4-*b*][1,4]benzothiazines (5-thiaisoalloxazines) are synthesized by reaction of 5-bromobarbituric acid with *o*-aminothiophenols (69IJC301). Synthesis of pyrimido[5,4-*f*][1,4]benzothiazepine are accomplished by Mannich-type thiazepine cyclization of 6-(*o*-aminophenylthio)uracil using aldehydes, such as formaldehyde and benzaldehydes (77S177). Pyrimido[4,5-*b*:5',4'-*e*][1,4]thiazine is obtained by heating 5,6-diamino-1,3-dimethyluracil and liquid hydrogen sulfide (89CPB2197) (Scheme 115).

A general transformation important for the synthesis of polycyclic heterocycles is the Smiles rearrangement of azaheterocycles. In the case of uracils, 6-chloro-5-nitro derivatives react with *o*-aminothiophenols. By such S → N rearrangements (71OR99; 73MI1), pyrimido[5,4-*b*][1,4]benzothiazines besides pyrimido[4,3-*b*]benzothiazolines are formed (74CPB1265; 80T2097) (Scheme 116).

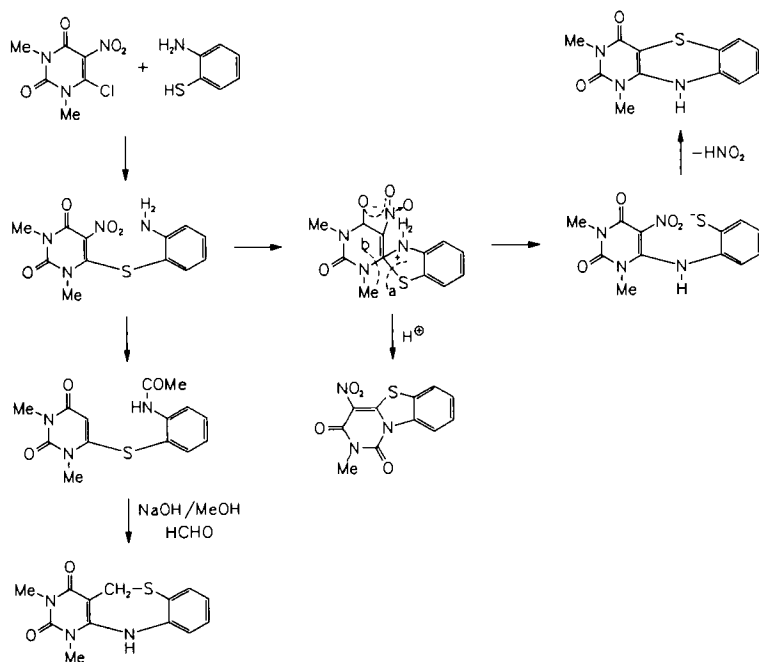
6-(*N*-Alkylanilino)uracils [72CC503, 72CPB1832, 72JOC4464; 73CPB-448; 74CPB1652; 76JA830; 77H(6)431, 77H(6)1179; 78H1767] and 6-(*N*-alkylanilino)-5-nitrouracils [75CC977; 76H(4)461; 77CC681, 77CPB563; 78H1767, 78JCS(P1)348] are used extensively as starting materials for the synthesis of isoalloxazines (flavins), isoalloxazine 5-oxides, and 8-chloroflavins. Isoalloxazines can also be synthesized from 5-amino-6-alkyl-anilinouracils [77H(6)25; 80CPB3576], 5-anilino-6-alkylaminouracils (84CC1691), and 6-anilino-5-nitroso uracil [71CPB206; 76H(4)461]. A simple synthesis of 10-arylisalloxazines involves condensation of 6-anilinouracils and nitrosobenzene [75JCS(P1)1907; 78H7; 79JHC1365; 88JCS(P1)313] (Scheme 117).



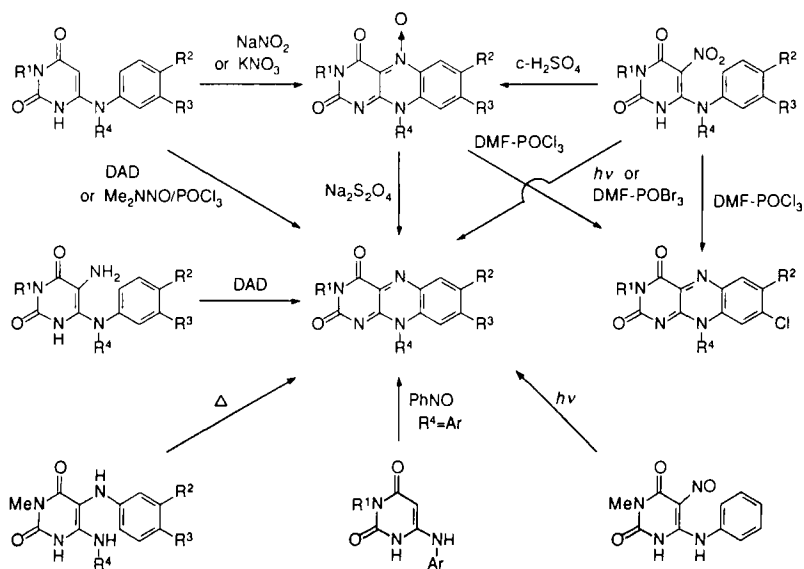
SCHEME 115

The 5-deazaflavin ring system a coenzyme model of oxidation–reduction systems, is formed by cyclization of 6-anilino-uracils using C_1 -reagents, such as Vilsmeier reagent (DMF- POCl_3) [76CC203, 76JCS(P1)1805; 82JHC929; 85JHC841], DMF-DMA [77H(6)1361], triethyl orthoformate (80CPB142), and carbon disulfide [81H(15)679]. Synthesis of the 5-deazaflavin starting from 6-chloro-5-formyluracils is described in Scheme 100. The reaction with trifluoroacetic anhydride gives 5-trifluoromethyl-5-deazaflavin (84AP42). The same class of compounds is obtained by exchanging the functional groups in both components (i.e., 2-chlorobenzaldehyde vs. 6-aminouracils) [88JCS(P1)313] (Scheme 118).

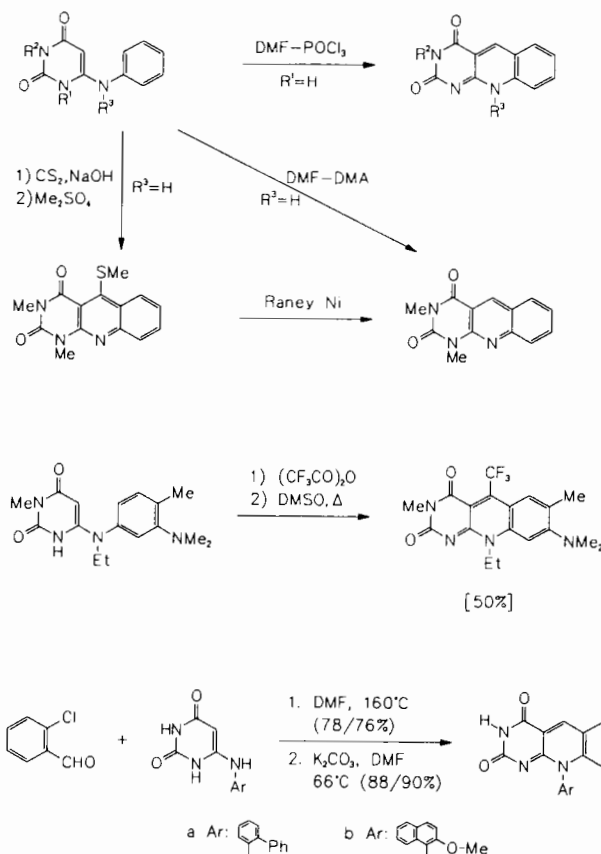
Analogous ring-closure of 6-aryloxy- and 6-arylthio-1-methyluracils yields 5-deaza-10-oxaflavins and 5-deaza-10-thiaflavins, respectively. The former oxidizes benzyl alcohol under neutral (aerobic) conditions



SCHEME 116



SCHEME 117

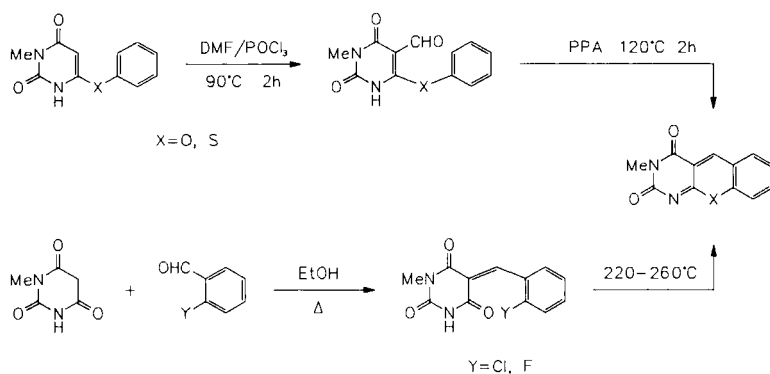


SCHEME 118

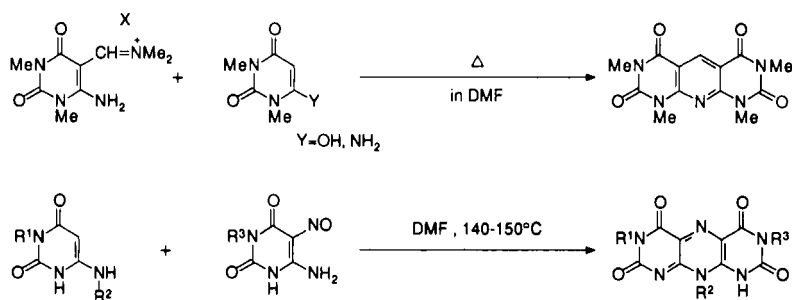
(80CL1157; 81JHC1329; 82JHC301). The 5-deaza-10-oxaflavin is also obtained by condensation of 1-methylbarbituric acid and *o*-halobenzaldehydes (90CPB307) (Scheme 119).

The Vilsmeier intermediate (see Scheme 58) reacts with 6-aminouracil or barbituric acid to give pyrido[2,3-*d*:6,5-*d'*]dipyrimidines (64CB1403; 85JHC345). Analogous methods for the formation of this ring system are known (71CPB1526, 71JOC1829; 83CPB344, 83S923). Pyrimido[5,4-*g*]pteridines are synthesized as flavin models (73CPB260; 83S563) (Scheme 120).

Another simple method for synthesizing 5-deazaflavins and 5-deaza-10-oxaflavins consists of a cyclization reaction of 6-chlorouracil with *o*-amino- and *o*-hydroxybenzyl alcohol, respectively (89CC44; 90CPB612) and also



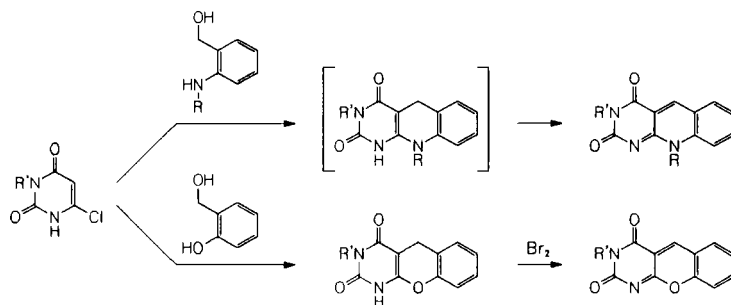
SCHEME 119



SCHEME 120

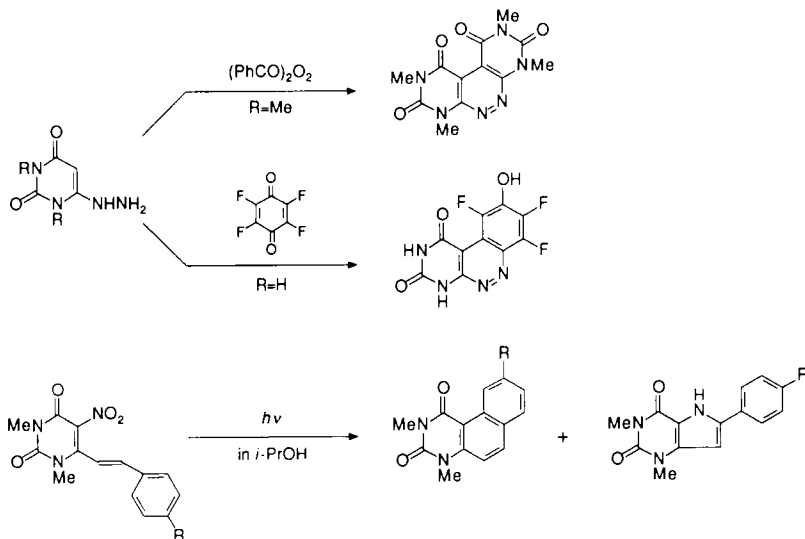
with anilines and subsequent Vilsmeier formylation (85JHC841) (Scheme 121).

6-Hydrazinouracils can be used to synthesize tricyclic pyrimidines. Thus, the reaction with benzoyl peroxide in acetic acid-chloroform and



SCHEME 121

with tetrafluoro-*p*-benzoquinone furnishes a pyridazino[3,4-*d*:6,5-*d'*]di-pyrimidine and a pyrimidol[4,5-*c*]cinnoline, respectively (74JMC1277; 79CPB2143). Tricyclic benzo[*f*]quinazolines are formed by photochemical reaction of 5-nitro-6-styryl-uracils in isopropanol [76H(4)461; 77CPB563] (Scheme 122).

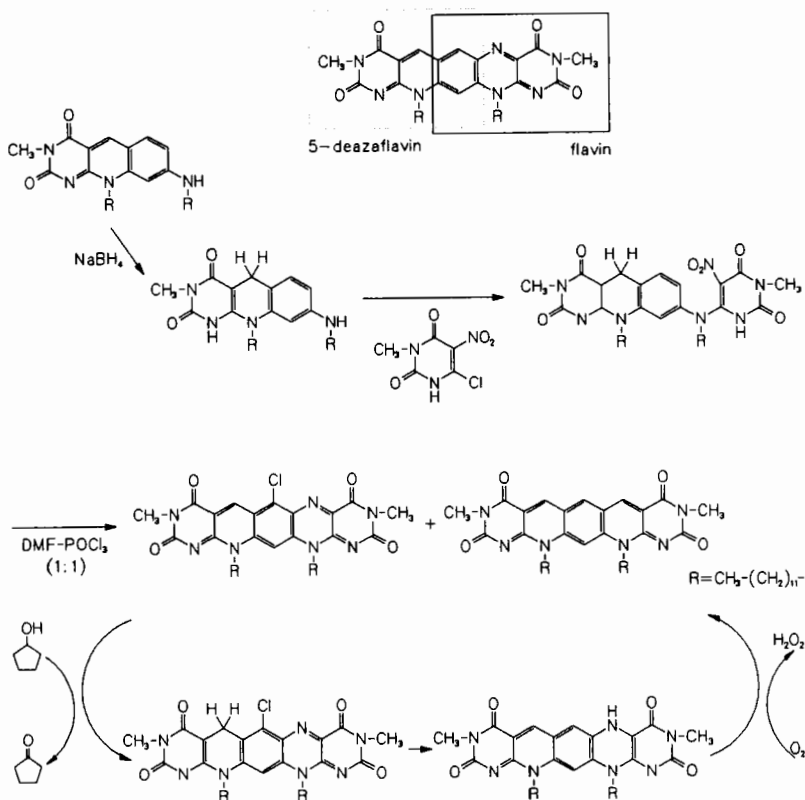


SCHEME 122

Mixed flavins [e.g., 1,3,7,9,11,12,14-heptaazapentacene-2,4,8,10-(3*H*,9*H*,12*H*,14*H*)-tetraones] oxidize alcohols in a neutral medium on sunlight irradiation. Therefore, they are of high biological interest. These compounds are composed of both a 5-deazaflavin and a flavin moiety. Their synthesis starts from an aminodeazaflavin then 1,4-reduction of the central pyridine ring and reaction with 6-chloro-5-nitouracil. Treatment with the Vilsmeier reagent leads to a pentacycle [84CC872, 84TL(25)5345; 88JCS(P1)1809, 88JHC549] (Scheme 123).

An angular 5-deazapteridino-desazaflavin with similar oxidative behavior is made from two molecules of 6-chlorouracil and 1,4-diaminobenzene with subsequent Vilsmeier cyclization (89CC44). Another approach to such a 5-deazaflavin is achieved in one step by reaction of two moles of 6-chloro-5-formyluracil with 1,4-diaminobenzene [86CPB2653; 88JCS(P1)1813; 89JHC49] (Scheme 124).

Uracil is 1-*N* alkylated by 2-chloromethylpyridine in the presence of NaH. This intermediate is cyclized by refluxing in phosphorus oxychloride to give a pyrido[1',2':3,4]imidazo[1,2-*c*]pyrimidinium system



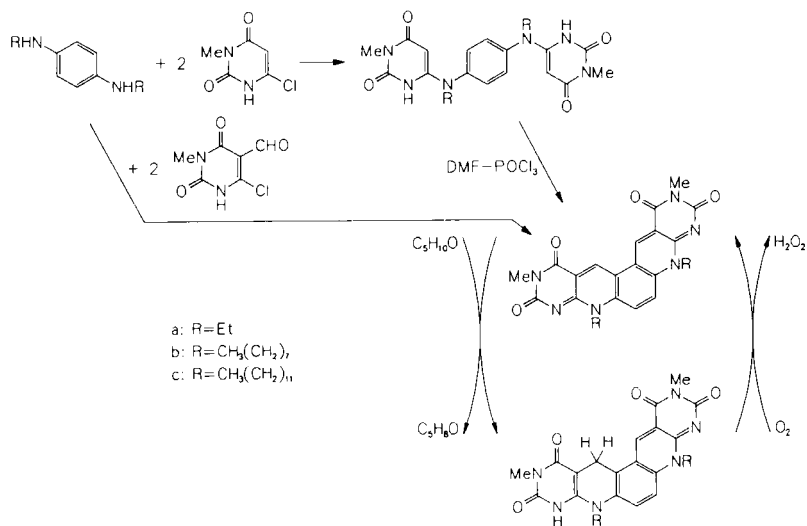
SCHEME 123

[87JCS(P1)2585]. *s*-Triazolo[4,3-*a*]purines are synthesized by condensation of ortho esters and 2-hydrazinopurine, which is readily available from 5,6-diamino-2-thiouracil (85CPB3113) (Scheme 125).

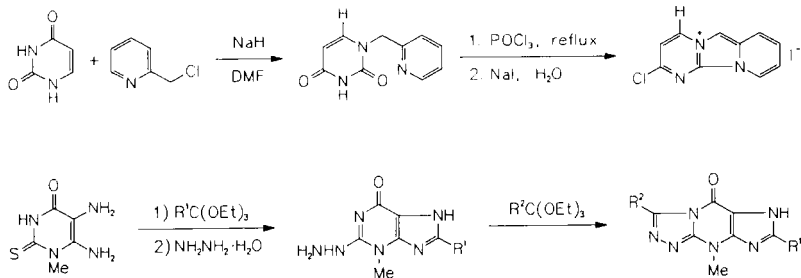
6-Aminouracil is cyclized by *N,N*-dimethylphosgeneiminium chloride and pyridine to give the tricyclic pyrido[1',2' : 1,2]pyrimido[4,5-*d*]pyrimidine-2,4-dione (81TL449; 83JHC575) (Scheme 126).

The 4-oxo group of 1-methyl-5-(2-bromoethyl)uracil is substituted by 1,2,4-triazoles using the system $\text{POCl}_3/\text{NEt}_3/\text{MeCN}$. Hydroxylamine hydrochlorides lead, in turn, to 3,4-dihydro-6*H*,8*H*-pyrimido[4,5-*c*][1,2]oxazine-7-ones (89H1735) (Scheme 127).

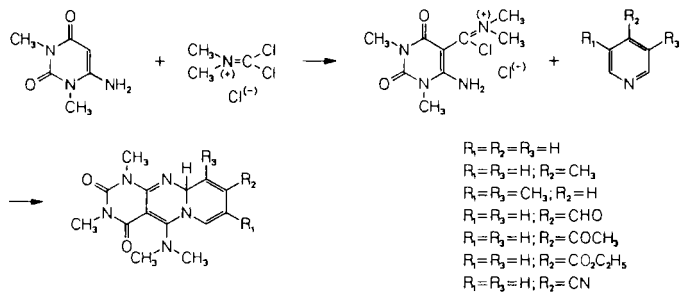
6-(Alkylthio)uracil-5-carbonitriles afford, with ethylene, diamine, hydrazine and hydroxylamine pyrimido[4,5-*b*][1,4]azepines, pyrazolo[3,4-*d*]pyrimidines, and isoaxazolo[3,4-*d*]pyrimidines, respectively (87JPR753) (Scheme 128).



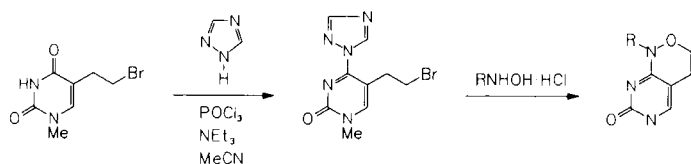
SCHEME 124



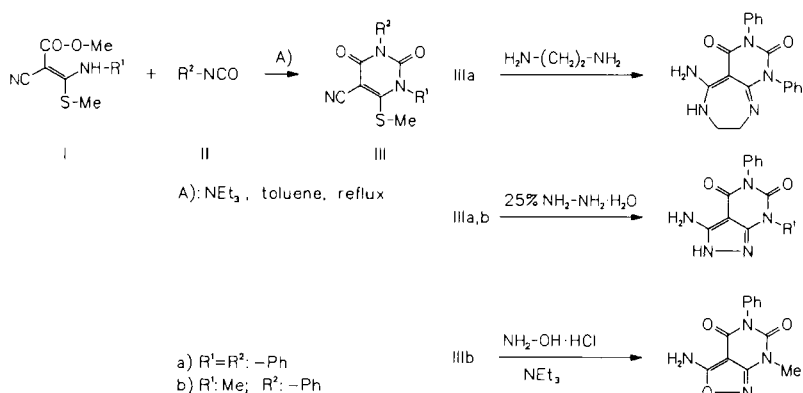
SCHEME 125



SCHEME 126



SCHEME 127

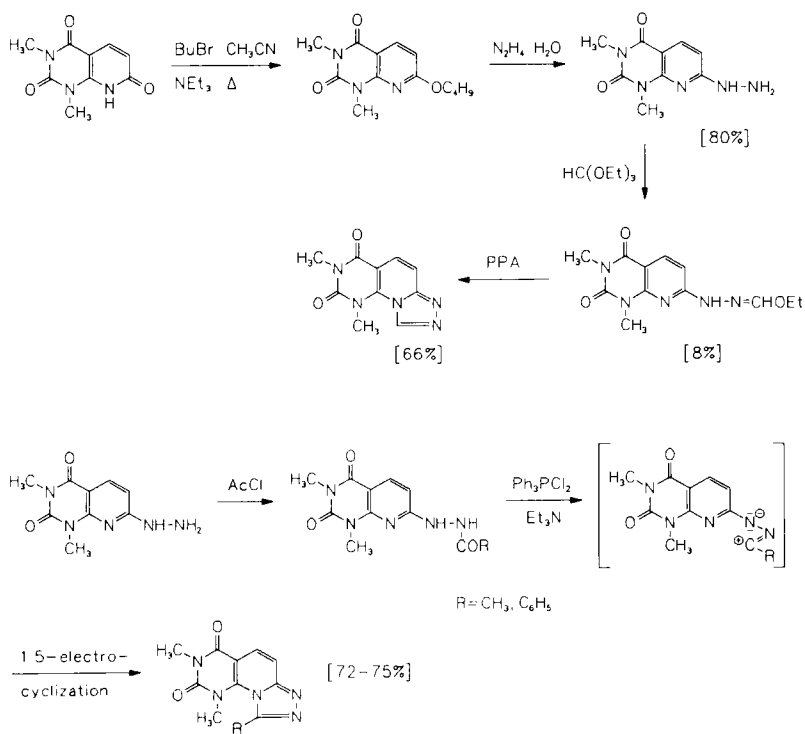


SCHEME 128

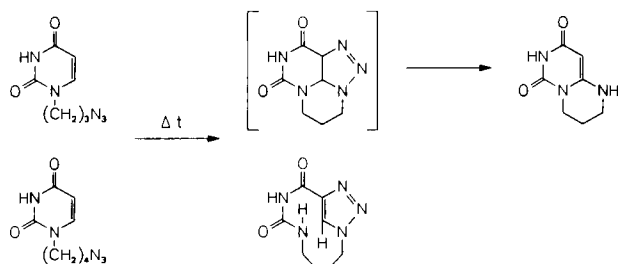
A simple synthesis of an *s*-triazolo[4',3':1,6]pyrido[2,3-*d*]pyrimidine derivative starts with 7-butoxypyridol[2,3-*d*]pyrimidine. Nucleophilic exchange with hydrazine and subsequent cyclization with a C-1 equivalent leads, in good yield, to the tricycle (84LA1653; 86CB943). The same ring system can be obtained elegantly by acylation of the hydrazine intermediate, followed by an intramolecular 1,5-electrocyclization by means of *in situ* prepared dichlorotriphenylphosphorane, a novel and simple access to the 1,3-dipole nitrilimine (87S876) (Scheme 129).

1-(3-Azidopropyl)- and 1-(4-azidobutyl)uracil give, after intramolecular 1,3-dipolar cycloaddition to the 5,6-double bond and subsequent denitrogenation, pyrimido[1,6-*a*]pyrimidines and 3,5,10,11,12-pentaazabicyclo[8,2,1]trideca-1,11-dienes, respectively (80T865) (Scheme 130).

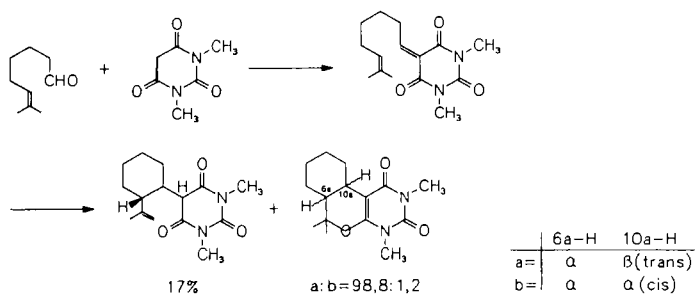
The reaction of 1,3-dimethylbarbituric acid with 7-methyl-6-octenal affords a Knoevenagel adduct that undergoes an intramolecular hetero Diels–Alder reaction to two diastereomeric tricyclic cycloadducts [90AG675, 90AG(E)665] (Scheme 131).



SCHEME 129



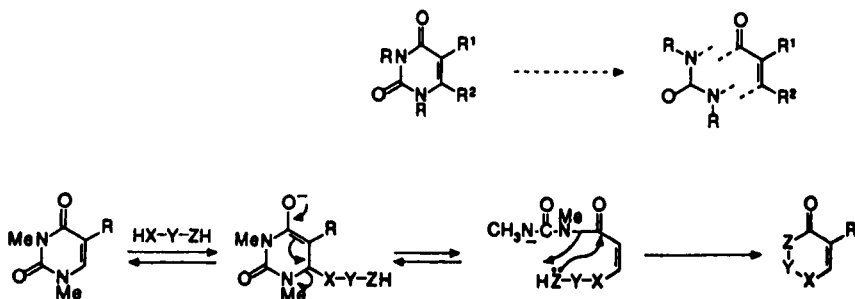
SCHEME 130



SCHEME 131

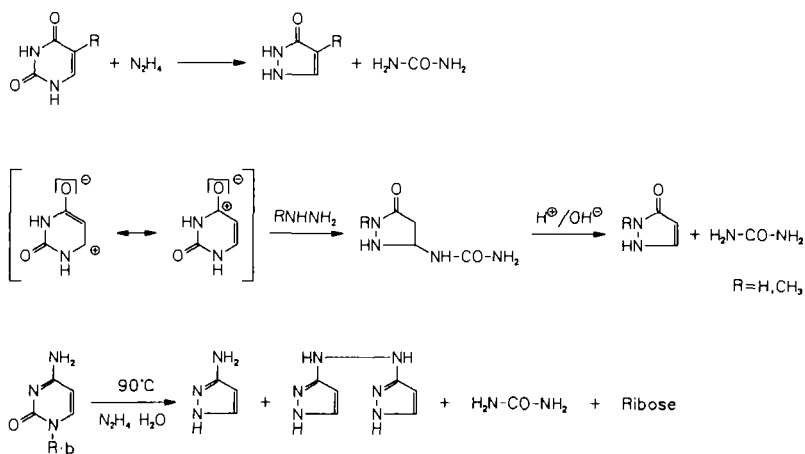
X. Dimethyluracil as Dimethylurea-Bridged Equivalent of α,β -Unsaturated Carbonyl Moieties—Ring Transformations

The aforementioned examples demonstrate a broad application of suitable substituted uracils for synthesizing numerous novel bi-, tri-, and oligoheterocyclic systems. Recently, an increasing number of examples have been presented which develop a new synthetic concept by cleavage of the uracil ring. Thus, various acyclic or cyclic 1,3-ambident nucleophiles cause an intramolecular ring transformation with displacement of the N(1)—C(2)—N(3) fragment of the uracil by another fragment of the nucleophile, e.g., by a N—N—C—, N—C—N—, C—C—N—, and C—C—C— fragment. This represents a preparatively valuable and versatile transfragmentation process as defined by Van Der Plas (73MI2) (Scheme 132).



SCHEME 132. Uracils as synthetic equivalents.

Pioneering work in this area took place as early as 1924, when it was shown that uracil and hydrazine give pyrazole derivatives with expulsion of urea (24CR811). Mechanistic studies followed 41 years later in which methylhydrazine and hydroxylamine were used as nucleophilic bases to produce pyrazolones and isoxazolones, respectively (64AG378; 65LA134; 82H2309). Another work reports the transformation of cytidine into an aminopyrazole and its *N,N*-dimers with simultaneous cleaving of the nucleoside bond [67JCS(C)1528]. This ring transformation is applied to the chemical modification of nucleic acid (59BCJ920, 59BCJ926, 59LA126; 64BBA462; 69BBA591) (Scheme 133).

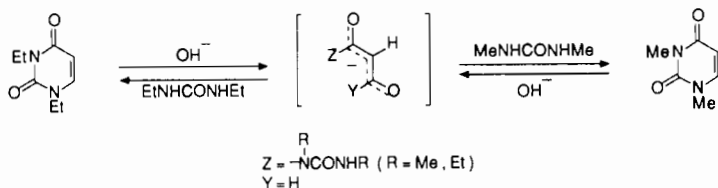


SCHEME 133

It is of biochemical interest that hydrazine and methylhydrazine cause mutations on microorganisms, and this can be used to produce biochemically deficient mutants [64ZN(B)151; 69MI2; 84ZPC1].

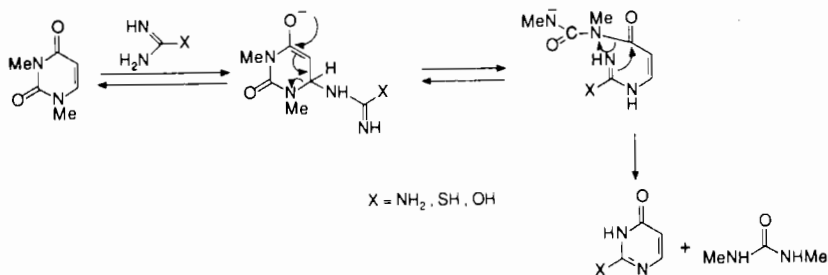
In contrast with 1,3-unsubstituted uracils, 1,3-disubstituted uracils are susceptible to various nucleophiles and can be regarded as a formylacetate masked with a 1,3-disubstituted urea. In fact, it is documented that 1,3-dialkyluracils in the presence of tetramethylammonium hydroxide behave as an enolate anion of formylacetate (77JOC2574; 78JOC3073) (Scheme 134).

1,3-Ambident nucleophiles, such as guanidine, thiourea, and urea attack 1,3-dimethyluracil first at the 6-position. After cleavage of the uracil ring and displacing ring closure, novel pyrimidines are formed extruding di-



SCHEME 134

methylurea, a pyrimidine-to-pyrimidine ring transformation (77JHC537; 78JOC1193; 83JOC3603) (Scheme 135).



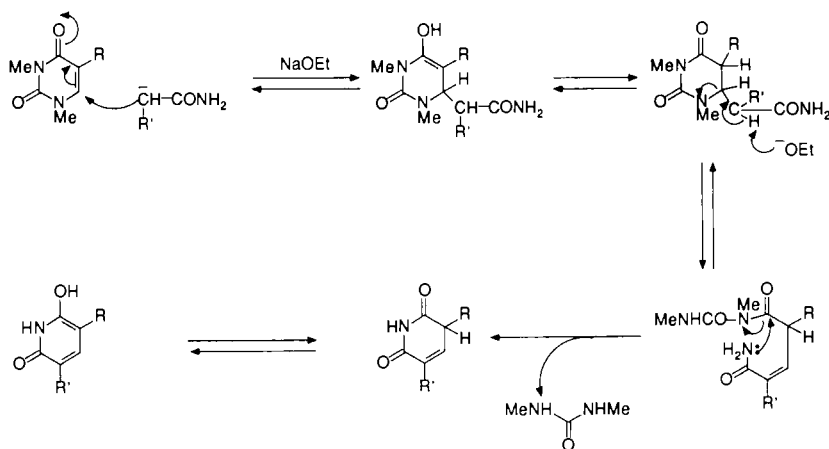
SCHEME 135

Instead of the nucleophilic N-atoms, the attempted attack of appropriate carbanions (from acetamides substituted with an electron-withdrawing group) on the C-6 position of dimethyluracil leads to the cleavage of the uracil ring. This reaction splits off dimethylurea and leads finally to the formation of tautomeric pyridine-2,6-diones, such as 2,6-dihydroxynicotinamide. Similarly, the reaction of 1,3-dimethyl-4-thiouracil with malonamide proceeds smoothly to give 2-hydroxy-6-mercaptopyridine-2,6-dione (79JA4423; 81JOC846) (Scheme 136).

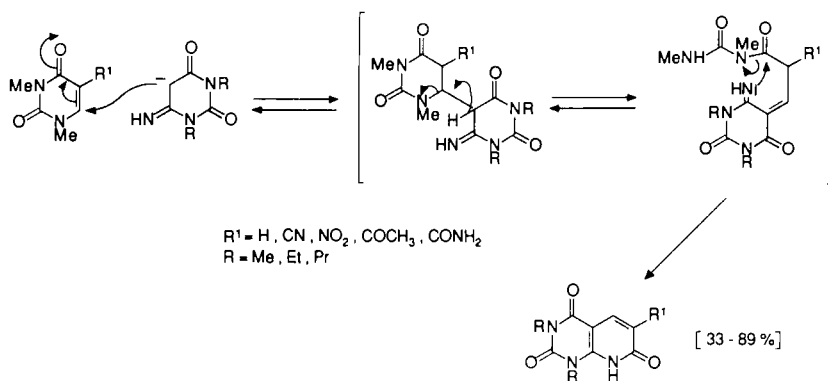
A cyclic 1,3-ambident nucleophile causes an analogous ring transformation to provide elegant access to pyrido[2,3-*d*]pyrimidines. This ring transformation is based on the initial nucleophilic attack of a 1,3-dimethyl-6-iminobarbiturate anion on the C-6 position of the uracil ring and on the easy displacement of the urea fragment with cyclization by the nucleophile in the presence of sodium ethoxide (80H407; 81JOC846) (Scheme 137).

The ring transformation of 5-nitrouracils using three carbon 1,3-ambident nucleophiles (for example, acetone) resulted in the isolation of the corresponding C6-adduct intermediates, which are easily converted into benzene derivatives (82T1405; 84JHC1543) (Scheme 138).

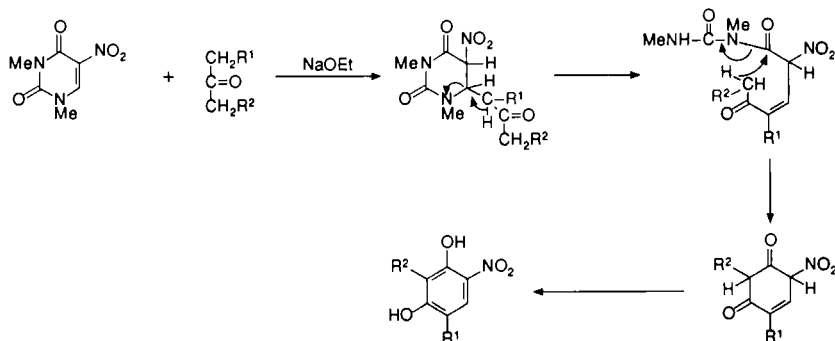
Similarly, the reaction of 5-cyano-1,3-dimethyluracil with the ambident nucleophiles acetone and acetonitrile exhibits an interesting transformation



SCHEME 136



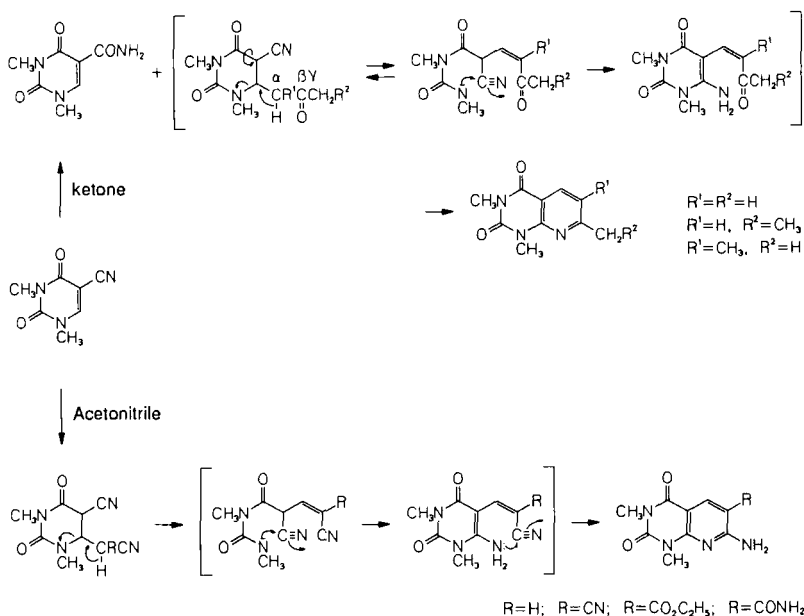
SCHEME 137



SCHEME 138

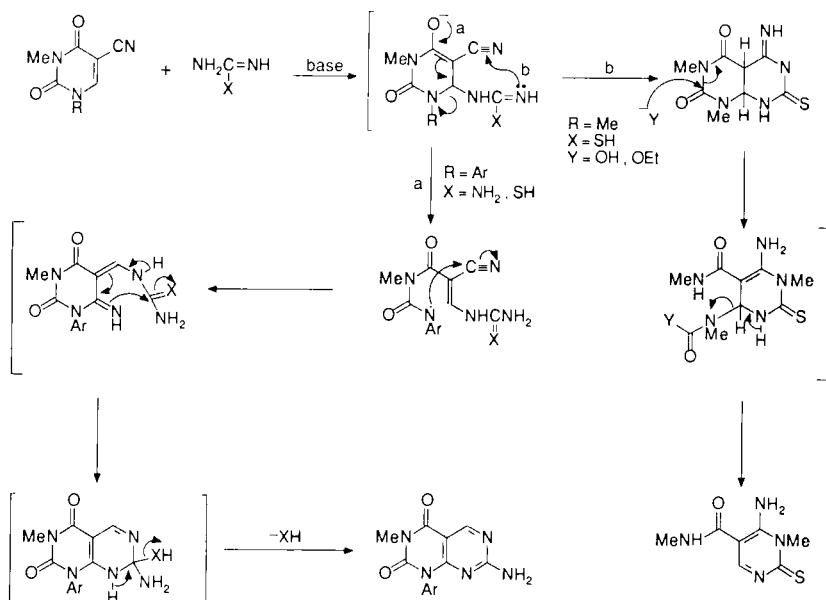
mechanism. With acetone, two products are obtained: 5-carbamoyl-1,3-dimethyluracil and a pyrido[2,3-*d*]pyrimidine. With acetonitrile, a Michael adduct is primarily formed, which then rearranges under ring cleavage into an open-chain intermediate. By a DOMINO-type double nucleophilic attack, pyrido[2,3-*d*]pyrimidines are formed (82JHC1261; 84JHC1543, 84MI3).

Such a ring cleavage and ring closure mechanism is supported by the rearrangement of 5-cyanouracils into 6-aminouracils in the presence of sodium hydroxide and amines (Scheme 139) (see also Scheme 143).



SCHEME 139

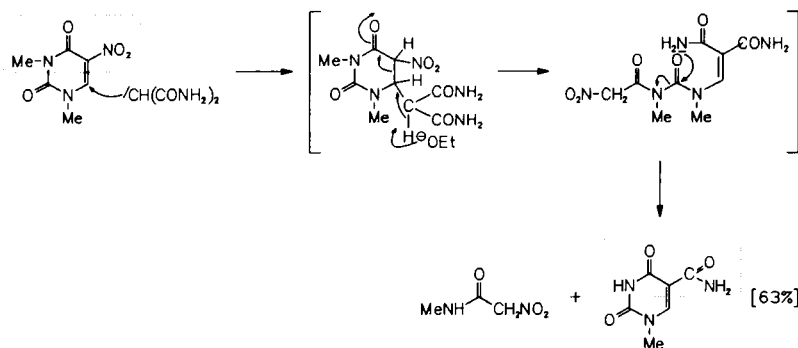
When thioureas are used as 1,3-ambident nucleophiles in the previous reaction, 1-phenyl-5-cyanouracils undergo analogous rearrangement and annulation to give pyrimido[4,5-*d*]pyrimidines. Reaction of 1-methyl-5-cyanouracils with thiourea and guanidines, however, induces another type of ring transformation to give 5-carbamoyl-2-thiocytosines and 2,4-diamino-5-carbamoylpyrimidines, respectively [84H2259; 85JOC1512; 90JCS(P1)123]. The difference in the reaction outcome can be explained by the fact that the substitution of a phenyl group at the N-1 position on the uracil ring remarkably facilitates the cleavage of the N-1—C-6 bond by attacking nucleophiles at the C-6 position (Scheme 140) (see also schemes 142 and 145).



SCHEME 140

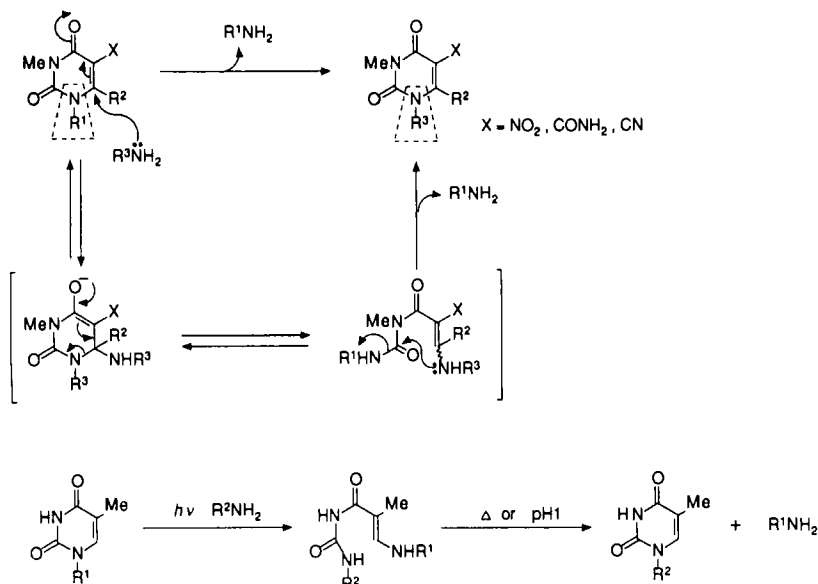
Another original transformation of a 5-nitrouracil into a 5-carbamoyluracil was observed on treatment of 1,3-dimethyl-5-nitrouracil with malonamide in ethanolic sodium ethoxide. In this case, the N(3)—C(4)—C(5) element of the uracil ring is replaced [81TL2409; 84JCS(P1)1859] (Scheme 141).

The reaction of 1,3-disubstituted uracils possessing an electron-withdrawing group, such as nitro, carbamoyl, and cyano at the 5-position with primary amines, results in the exchange of the N-1 portion of the uracil



SCHEME 141

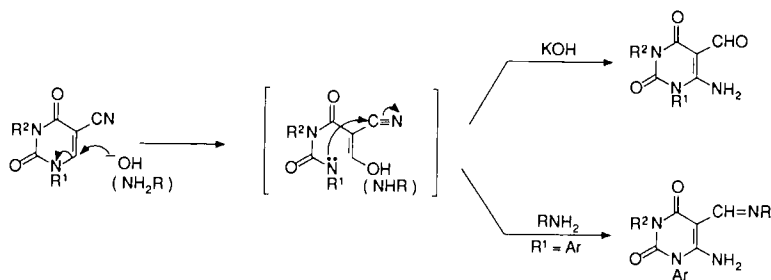
ring with the amine moiety. The exchange reactions are influenced by the 5- and N-1 substituent. Substitution of an N-1-phenyl group promotes the present reaction. The reaction mechanism is explained in terms of addition, ring opening, and ring-closure [86TL3263; 90JCS(P1)367]. Photochemical reaction of thymine derivatives with alkylamines induces an exchange reaction apparently similar to the previous one (81JA1598, 81TL3265). The photochemical reaction involves an initial nucleophilic attack of amines at the 2-position of the photoexcited thymine, as evidenced by isolation of the intermediate urea, and is applied to the chemical modification of nucleic acids (83JA956, 83JA6989) (Scheme 142).



SCHEME 142

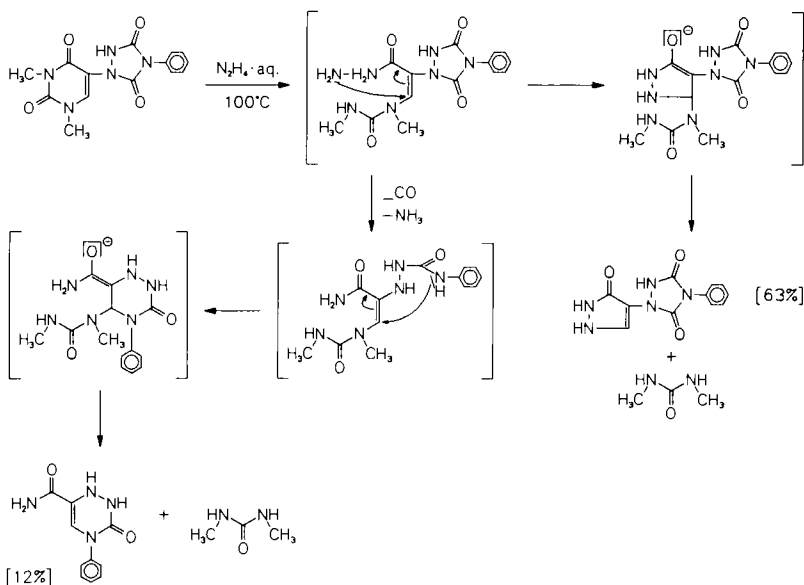
In the presence of potassium hydroxide, 5-cyanouracils undergo a Dimroth-type rearrangement involving a ring-opening and ring-closing process to give 6-amino-5-formyluracils. Similar ring transformation is observed in the reaction with ammonia and primary alkylamines (84H2259; 89CPB2008) (Scheme 143).

A special case of these ring transformation reactions is the 5-insertion reaction of 4-phenyl-1,2,4-triazolin-3,5-diones (4 Ph-TAD) [75OPP251; 81AG832, 81AG(E)797] into uracils, for example, the reaction of 5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-1,3-dimethyluracils with hydrazine



SCHEME 143

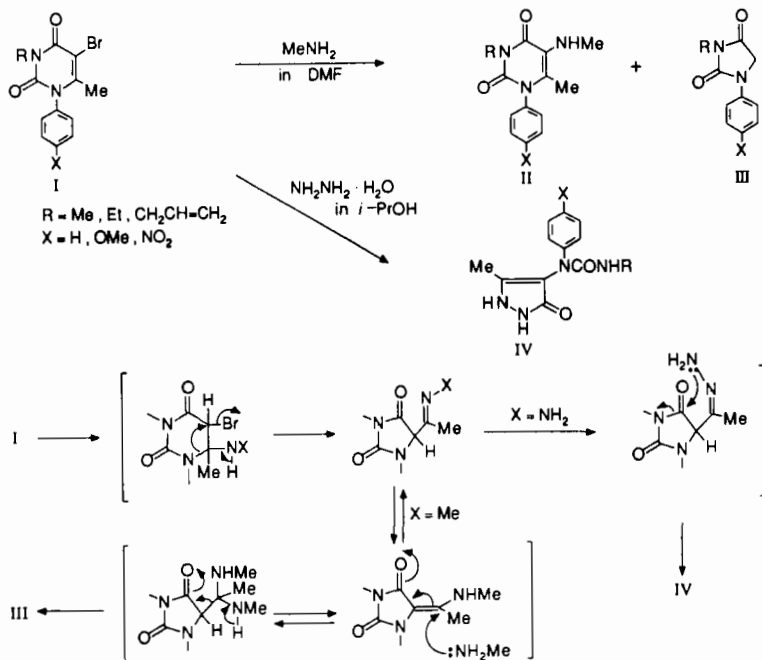
hydrate. Depending on whether only the uracil ring undergoes cleavage or both the uracil and the 1,2,4-triazolidine rings are cleaved, pyrazolone-4-yl-1,2,4-triazolidines and a 4-oxo-1,2,4-triazine-6-carboxamide have been obtained (77CB1716) along with dimethylurea (Scheme 144).



SCHEME 144

When 5-bromo-6-methyluracils possessing a phenyl group at the N-1 position are treated with alkylamines and hydrazine, ring contraction occurs to hydantoin and 3-pyrazolones, respectively. The latter conversion into the pyrazolone is a double-ring transformation via a hydantoin intermediate. The N-1-phenyl group is essential for the ring contraction to

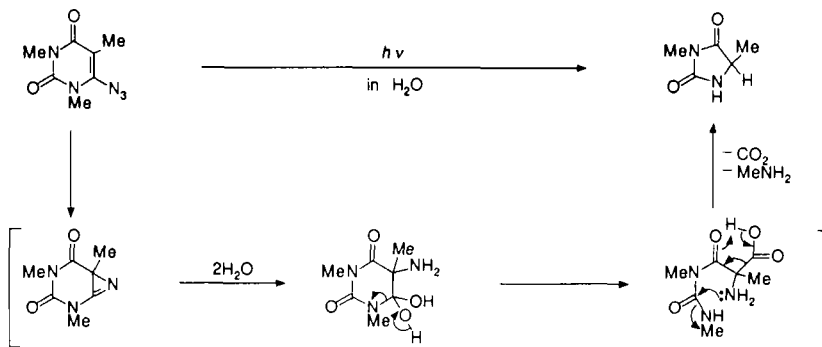
occur. A detailed mechanism has been proposed [74TL3087; 84H2309; 85JCS(P1)1137]. Transformation into a hydantoin ring system is frequently observed with fused uracil derivatives, such as flavin [67JOC3049; 77CC175; 84TL(36)4015], lumazine (73TL1681; 74CJC3879; 76T2121), and pyrimido[5,4-*b*][1,4]thiazine (76CPB3135) (Scheme 145).



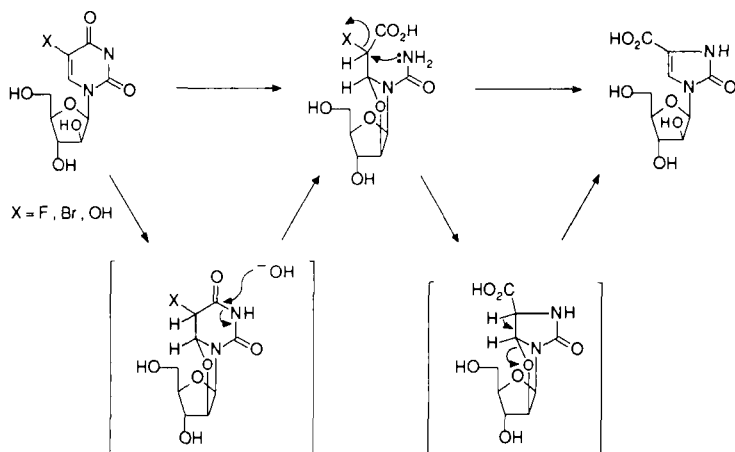
SCHEME 145

The ring contraction of uracil into hydantoin also occurs in the photochemical reaction of 6-azido-1,3,5-trimethyluracil in water, although in general, irradiation of 5-substituted 6-azidouracils in the presence of amines induced ring expansion to 1,3,5-triazepines (see Scheme 109). Both reactions proceed by a common azirine intermediate. Thus, bimolecular addition of water to the azirine gives 5,6-dihydrouracil, which undergoes cleavage of the N-1—C-6 bond, recyclization, and subsequent decarboxylation to give rise to the hydantoin [84JCS(P1)1719] (Scheme 146).

Reaction of 5-halogeno (or hydroxy) arabinofuranosyluracils in alkaline media causes ring transformation into imidazoline nucleosides involving neighboring-group assistance of the 2'-hydroxy group (67JA3663; 68JOC3593, 68TL2967; 69JOC1390, 69JOC2636) (Scheme 147).



SCHEME 146

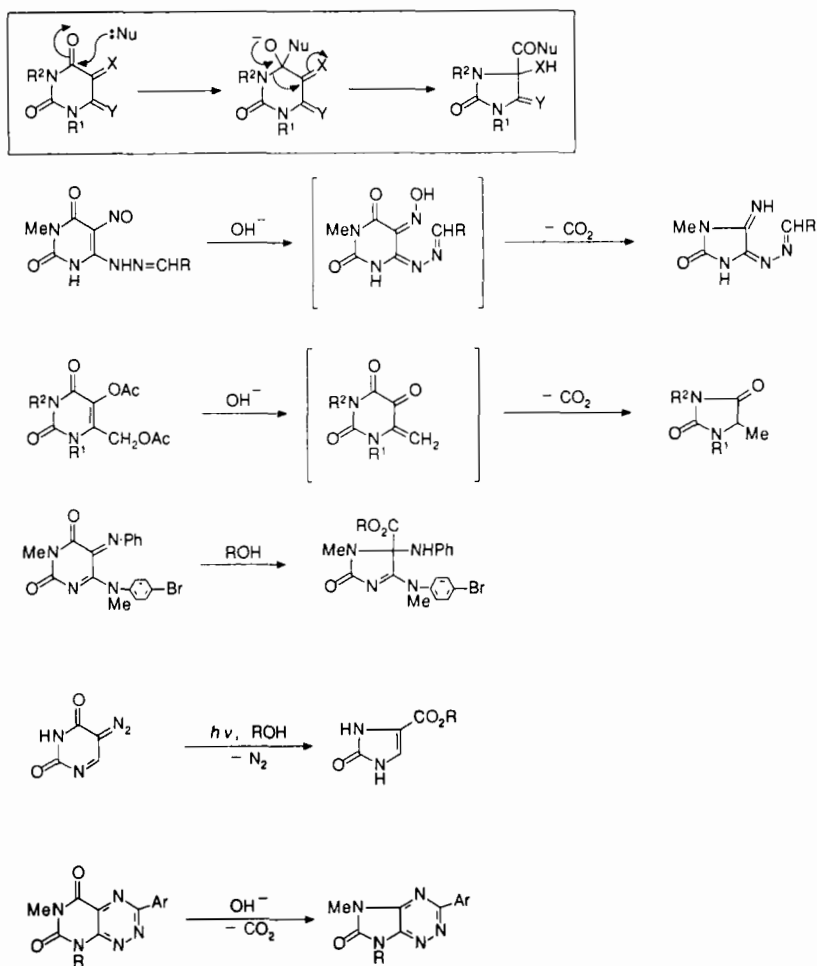


SCHEME 147

Analogous ring transformations are observed in the reaction of 6-benzylidene-hydrazino-5-nitrosouracil (80H1295), 5-acetoxy-6-acetoxymethyluracil (82JOC508; 83JHC753; 86MI2), 5-phenylimino-6-(*N*-methylanilino)uracil (84H2509), 5-diazo-uracil (79H761), and 7-azapteridine [76H(9)1503]. Here a hetero-diene structure is included in starting compounds or reaction intermediates (Scheme 148).

An adduct of 1-substituted 5-diazo-uracils and methanol undergoes ring contraction in the presence of water to give 1,2,3-triazoles (73JA3081; 74JHC645; 76JOC1041; 77JHC647; 78JHC1349) (Scheme 149).

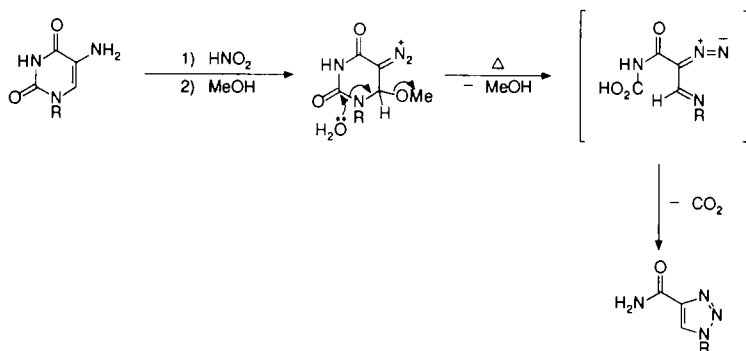
Another promising uracil transformation reacts ambident C—C—C nucleophiles with 5-formyl-1,3-dimethyluracil. With various methylene car-



SCHEME 148

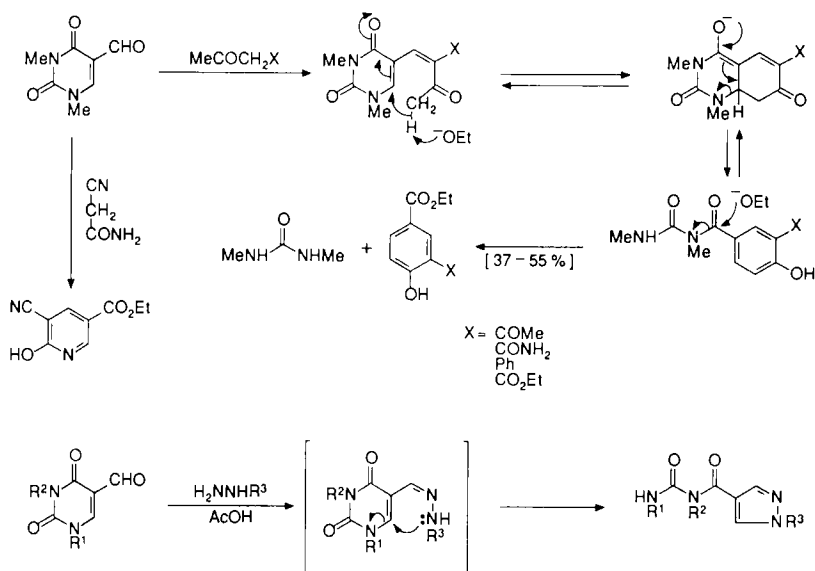
bon acids, an additional process is observed: transformation of heterocyclic to carbocyclic compounds, for example, the formation of benzene derivatives. When cyanoacetamide, a $C-C-N$ type of nucleophile, was used as the active compound, ethyl 5-cyano-6-hydroxynicotinate was obtained in 46% yield (80JHC413; 81JOC3949; 82H185).

Although hydrazinolysis of uracil derivatives into pyrazoles is well known (see Scheme 133), reaction of 5-formyluracils with hydrazines in the presence of acetic acid causes another type of ring contraction



SCHEME 149

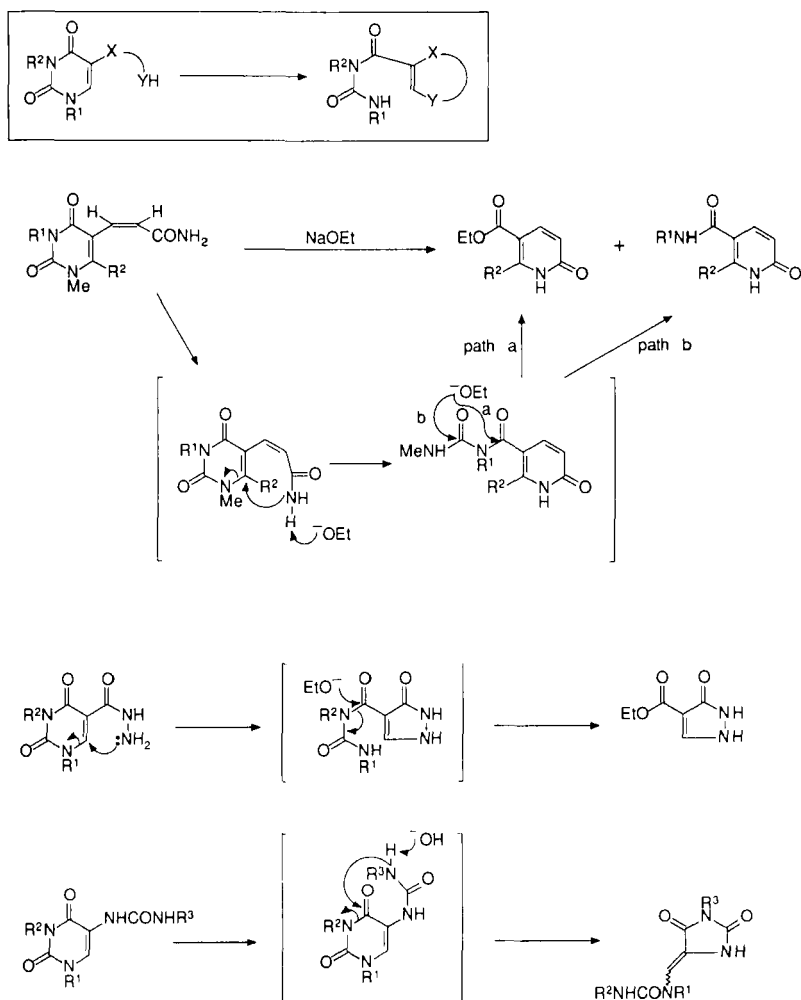
similar to the reaction mode just described [68JOC892; 81CPB3760; 83JCS(P1)1293] (Scheme 150).



SCHEME 150

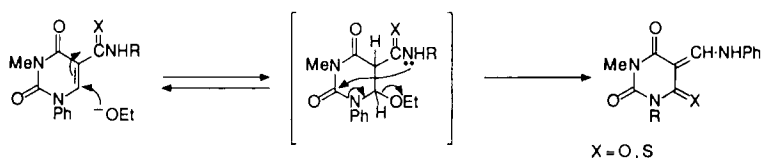
The mechanism shown in Scheme 150 suggests that the presence of a terminal nucleophile in the sidechain on a uracil ring enables the ring transformation of uracil into other rings by an intramolecular rearrangement. In fact, in the presence of sodium ethoxide 5-(2-carbamoylvinyl)uracil-5-carbohydrazides are easily converted into pyridines and 3-

pyrazolones, respectively, by an intramolecular attack on position 6 [85JOC1512; 92(ip)]. Similar reaction of 5-ureidouracils, however, furnishes 5-ureidomethylenehydantoins resulting from an attack on the 4-carbonyl carbon, due to a difference in the electronic nature of the 5-substituent. In contrast with other 5-substituents, the 5-ureido group has electron-donating character and reduces the reactivity of the 6-position toward nucleophiles [92JCS(P1)(ip)] (Scheme 151).



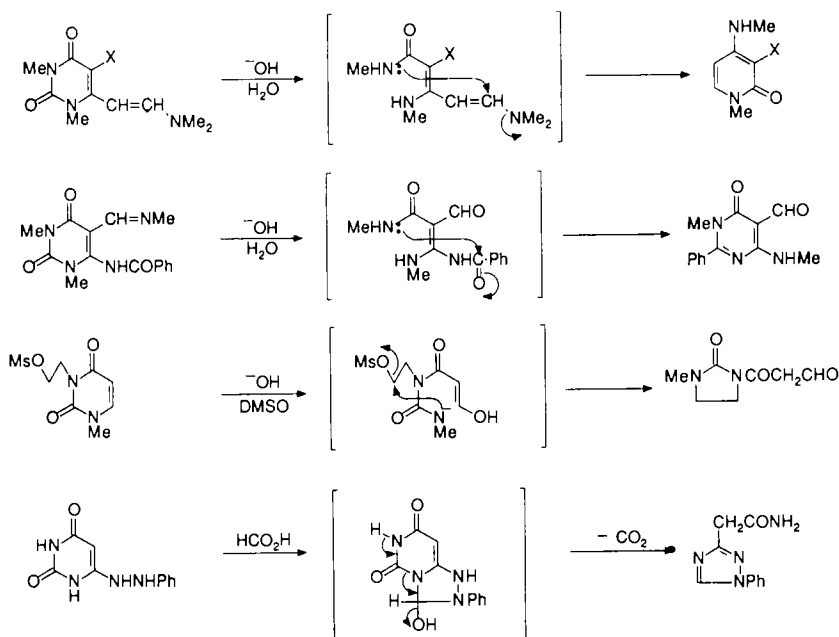
SCHEME 151

Reaction of 5-carbamoyl- and 5-thiocarbamoyl-3-methyl-1-phenyluracil in ethanolic ethoxide induces an intramolecular rearrangement to give barbituric acids and 4-thiobarbituric acid, respectively, by a nucleophilic attack of the 5-carbamoyl group on the 2-carbonyl group [81MI1; 89JCS(P1)1695; 90T3431] (Scheme 152).



SCHEME 152

Rearrangement occurs to afford a new ring on hydrolysis of uracil derivatives possessing an appropriate sidechain. Thus, alkaline hydrolysis of 6-dimethylaminovinyluracils, 6-benzoylaminouracil, and 3-(2-methanesulfonyloxyethyl)uracil leads to the formation of 2-pyridones, 4-pyrimidone, and 2-imidazolinone, respectively (68CB512; 75JOC1722; 78H739; 88JHC985). On heating in formic acid, 6-phenylhydrazinouracil undergoes a ring transformation to give a 1,2,4-triazole (77JHC701) (Scheme 153).

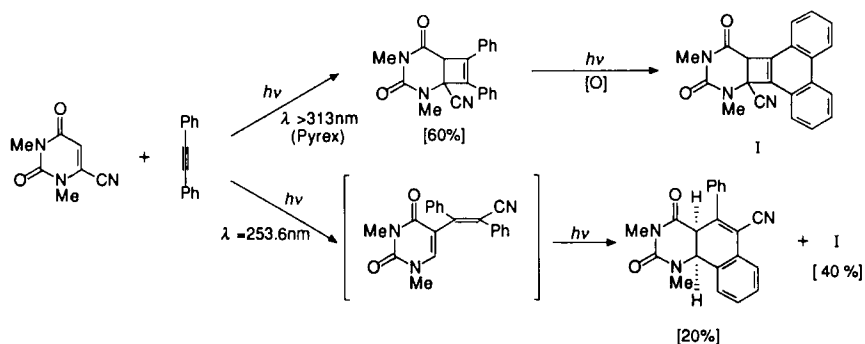


SCHEME 153

XI. Photoinduced Electrocyclizations and Radical Reactions of 1,3-Dimethyluracils

1,3-Dimethyluracil-6-carbonitrile gives, on UV irradiation (pyrex-filter; $\lambda > 313$ nm) with tolane (diphenylacetylene), cyclobutene adducts which subsequently undergo 6π -electron cyclization (photophenanthrenization) on additional irradiation to afford pentacyclic systems containing a uracil moiety.

Short wavelength irradiation (quartz filter; $\lambda = 253.6$ nm) results in 1,4-migration of the nitrile function. The resulting vinyluracil undergoes 6π -electron cyclization (80JA3948, 80TL2317) (Scheme 154).

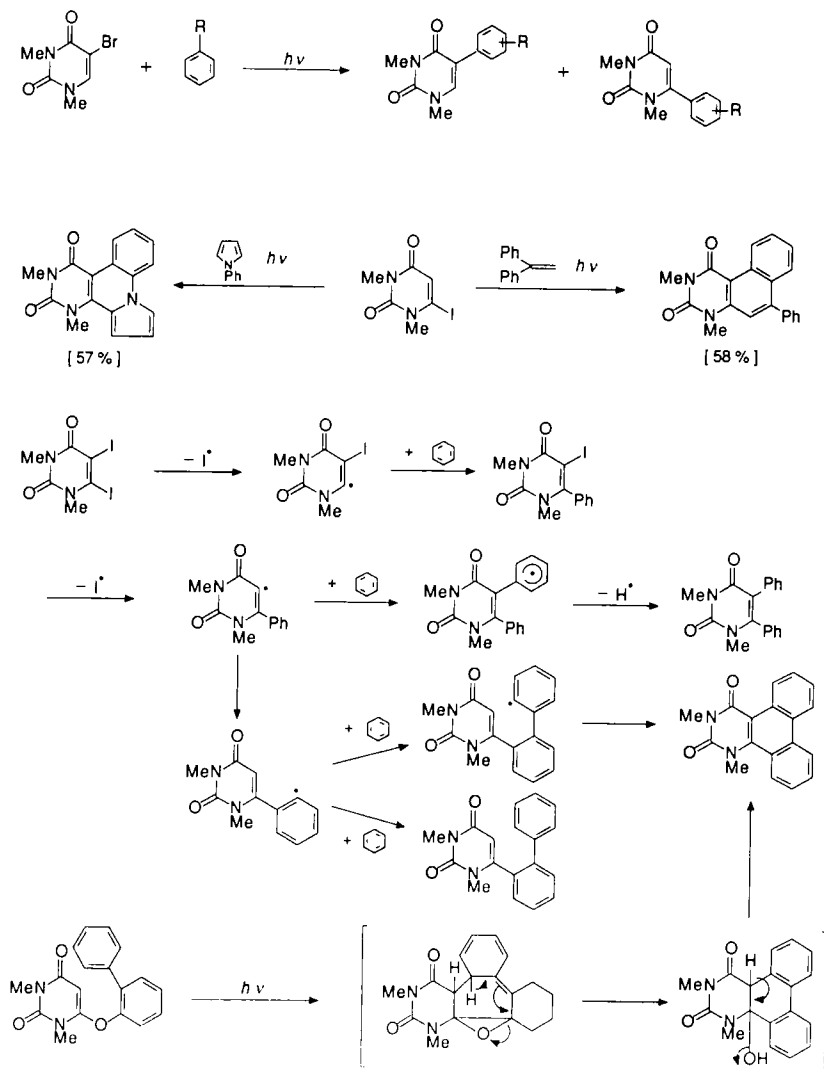


SCHEME 154

Irradiation of 5-bromo (iodo) uracils in benzene induces coupling with the solvent to give 5- and 6-phenyluracils (73TL4317; 86CL195; 87CL175). 6-Iodouracils and 1,1-diphenylethylene or 1-phenylpyrrol photocouple to give fluorescent uracil derivatives (85TL1743).

A radical mechanism is also proposed when 5,6-diiodouracil is irradiated in benzene to lead to a mixture of 5-iodo-6-phenyluracil, 5,6-diphenyluracil, 6-bi-phenyl-2-yl-uracil, and finally dibenzoquinazoline [74JCS(P)2649]. The dibenzo-quinazoline is also formed by photocyclization of 6-*o*-biphenyloxyuracil (74JA315) (Scheme 155).

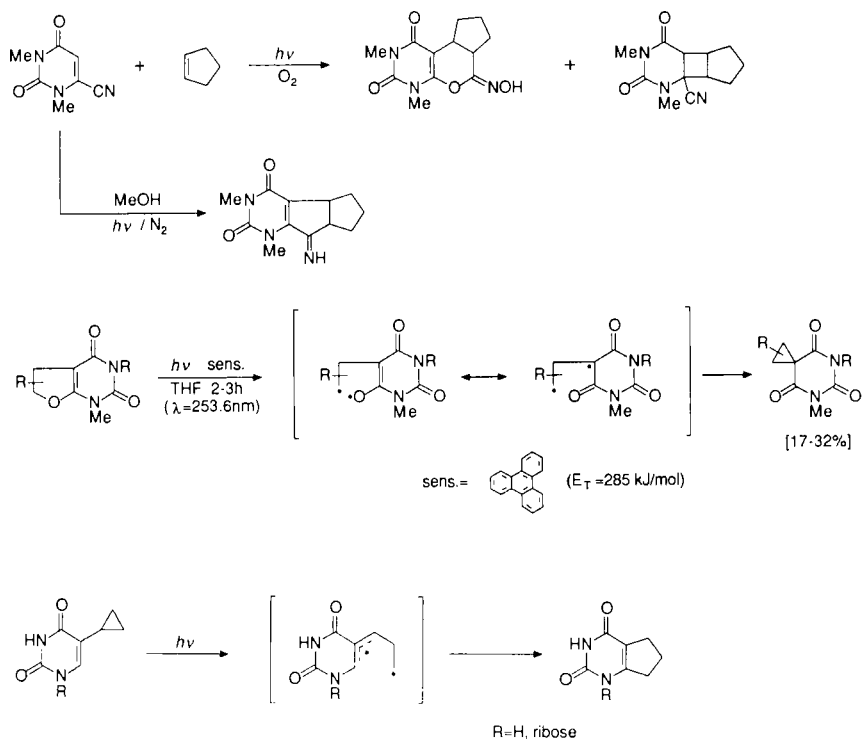
In the course of many photoreactions, diradical species form normally as intermediates; in special cases, these can be intercepted by suitable scavenger molecules. Oxygen trapping reactions of these 1,3-biradical intermediates formed in the photoaddition of 2-cyanochromone or 6-cyano-1,3-dimethyluracil to olefins have been described. The latter reacts under $[3 + 2]$ cycloaddition (83JA963, 83TL2195). Furo[2,3-*d*]pyrimidines cleave upon sensitized UV irradiation to give, via diradical species, 5,7-diazaspiro[2,5]octanes (87CB1433). 5-Cyclopropyluracil rearranges to the



SCHEME 155

5,6-trimethyleneuracil by a biradical intermediate (76TL4379) (Scheme 156).

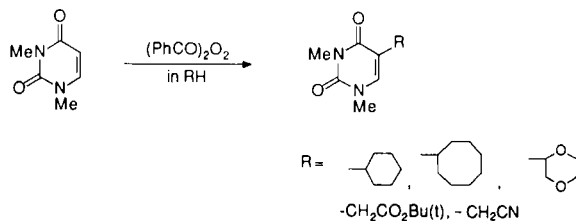
On heating 1,3-dimethyluracil in cyclohexane in the presence of benzoyl peroxide, 5-cyclohexyl-1,3-dimethyluracil is formed via a free radical alkylation. Similar regioselective alkylation proceeds in cyclooctane, diox-



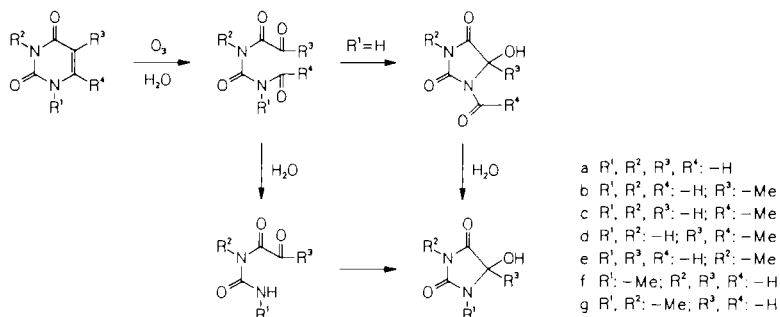
SCHEME 156

ane, *t*-butyl acetate, and acetonitrile to yield the corresponding 5-alkyluracils (89CL977) (Scheme 157).

Ozonolysis of cellular substances, such as uracil and thymine derivatives, is of special interest. Ozonolysis of aqueous solutions of uracils leads to ring cleavage and subsequent ring contraction forming 5-hydroxyhydantoin (89CL723; 90JOC1396) (Scheme 158).

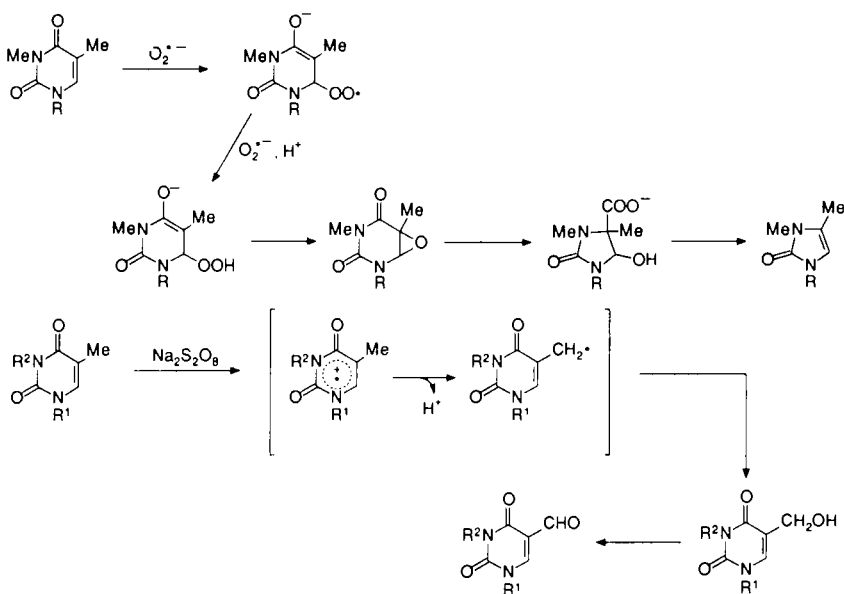


SCHEME 157



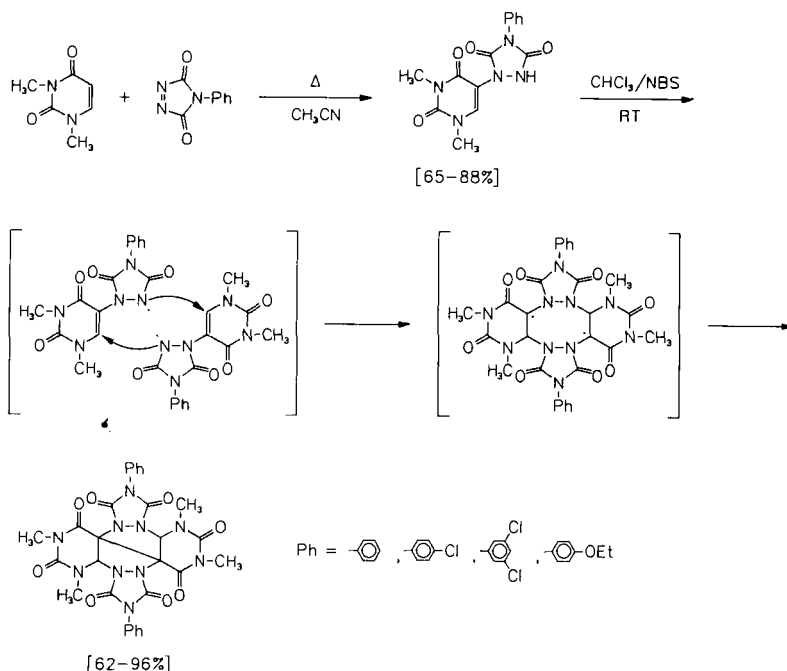
SCHEME 158

The oxidation of thymine and thymidine derivatives with superoxide ion in dimethyl sulfoxide (DMSO) or DMF gives ring-contracted imidazolones (88CC1171). Oxidation with sodium persulfate gives a mixture of 5-hydroxymethyluracils and 5-formyluracils, perhaps by thymine cation radicals (86CL1319; 88JOC3421) (Scheme 159).



SCHEME 159

These aforementioned 5-(1,2,4-triazolidin-1-yl)uracils (77CB1716) are easily oxidized by NBS to urazolyl radicals (77CB1699), which attack each other head-to-tail to form tetrazocane diradicals that then recombine in the final step to afford bridged 1,2,5,6-tetrazocanes with uracil and urazolo bridges (X-ray). This is a decisively simple approach to a rather complex heterocyclic molecule in one step (85CB436) (Scheme 160).



SCHEME 160

XII. Novel Cleavage and Transformation Reactions of 6-Aminouracils

Section X shows how the attack of ambident nucleophiles, preferably on the C-6 position of uracils, initiates a general fragmentation process leading to several novel and interesting heterocycles. Treatment of 6-aminouracils with dialkyl acetylenedicarboxylates in protic media leads to Michael addition at C-5 with subsequent cyclization to the aforementioned pyrido[2,3-*d*]pyrimidines, which possess interesting biological (antitumor) activity (72CL657, 72JOC578; 73CPB2014; 76JOC1095). However, this

These surprising results force revision of some papers that appeared in this field discussing other products that were assigned, such as a 6-amino-5-(3-carbomethoxy-2-propynoyl)-1,3-dimethyluracil structure (72CL657, 72JOC578; 77JOC4159; 82CPB63).

This unique addition–transformation process has also been found in cycloaddition of acetylenic esters to dienes and azadienes containing the 5,6-double bond of uracil as part of the chromophore. Activated and polarized dienes of this type afford, with olefines, pyridol[2,3-*d*]pyrimidines and quinazolines products of a polar [4 + 2]-cycloaddition followed by subsequent aromatization by elimination of dimethylamine or followed by oxidation on heating in nitrobenzene. Azodicarboxalates afford Michael adducts, which are thermally converted into 8-(di-methylamino)theophylline.

However, treatment with dimethyl acetylenedicarboxylate gives only, in one example, the expected pyrido[2,3-*d*]pyrimidine; in all other cases the previously mentioned Michael-addition–ring cleavage–cyclization–condensation sequence is observed to afford pyrrolo[3,4-*c*]pyridine-diones (cf. Scheme 128) (88TL4401; 89CB1673) (Scheme 163).

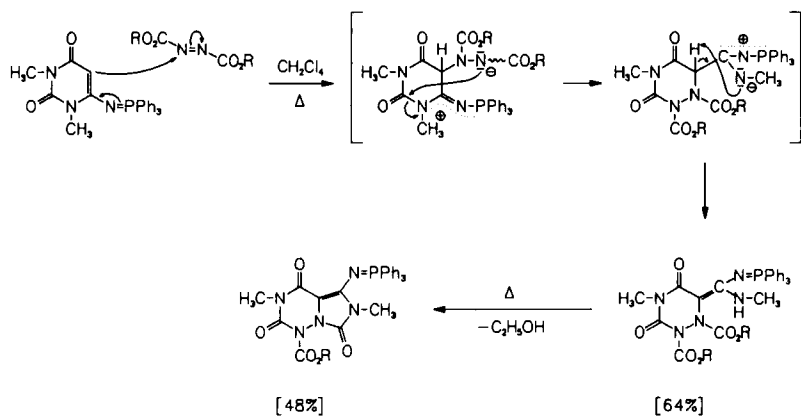
This novel transformation is also observed with diethyl azodicarboxylates and 6-(triphenylphosphoranylideneamino)-1,3-dimethyluracil to give a 6-amidinylidene-3,5-dioxo-1,2,4-triazine-1,2-di-carboxylate, which can then be thermally cyclized into imidazo[5,1-*f*][1,2,4]triazine. Some of these bicyclic heterocycles are known to be biologically active (86JOC2787) (Scheme 164).

5-Formyl-1,3,6-trimethyluracil is transformed by a [1,5-*H*]-shift into a 5-hydroxy-methylene-6-methyleneuracil intermediate, an *o*-quinodimethanes that is capable of [4 + 2]-cycloadditions with aldimines of the same uracil to afford pyrido[3,4-*d*]pyrimidines in a one-step preparation. Accordingly, maleimide leads to pyrrolo[3,4-*g*]quinazolines (88JHC205; 89PC1) (Scheme 165).

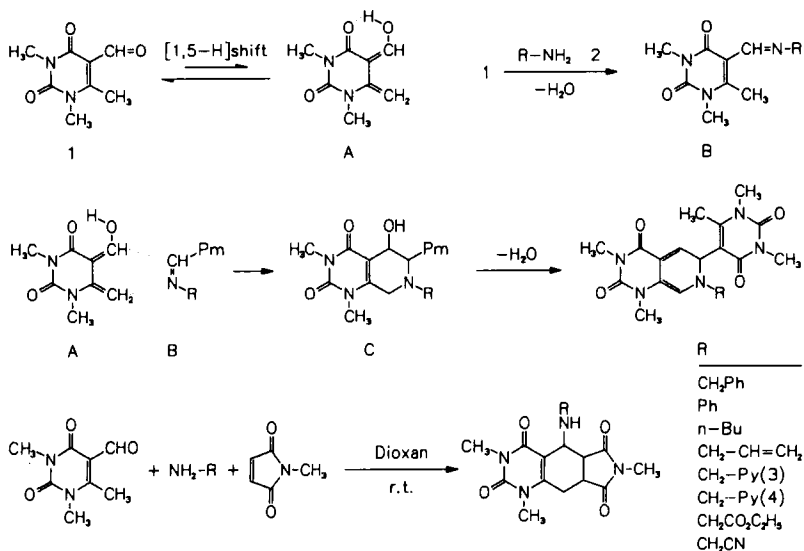
5-Formyl-6-aminomethyluracil derivatives give imines with primary amines. These are in equilibrium with a 5,6-bisaminomethylene-uracil. Internal cyclization gives pyrrolo[3,4-*d*]pyrimidines (89BCJ3043) (Scheme 166).

The intermediate 5,6-dimethyleneuracil tautomer of 5-formyl-6-methyluracils react with aldimines to give pyrido[3,4-*d*]pyrimidines, which can be converted into 2,4,8-trioxopyrido[3,4-*d*]pyrimidines (89MI1) (Scheme 167).

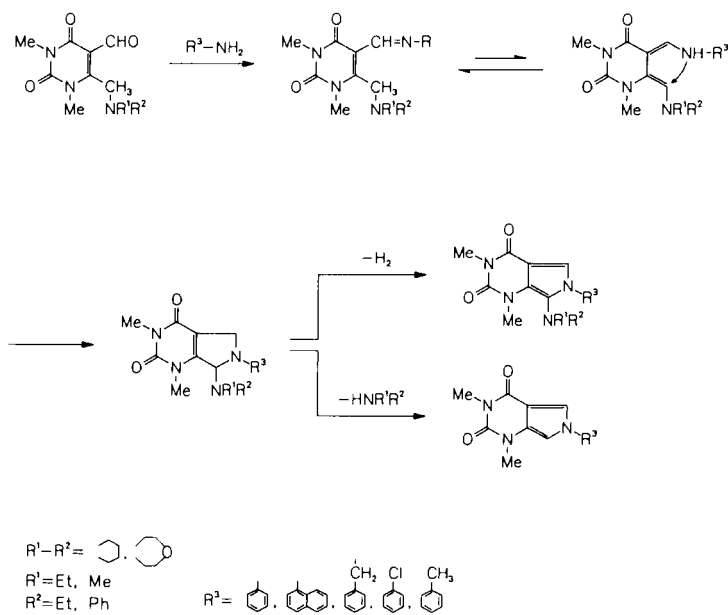
At temperatures of ~130–200°C, 6-alkylamino-5-vinyluracils undergo [1,5]-sigmatropic *H*-migration, as revealed by deuteration experiments (85H2057; 86CB943) (Scheme 168).



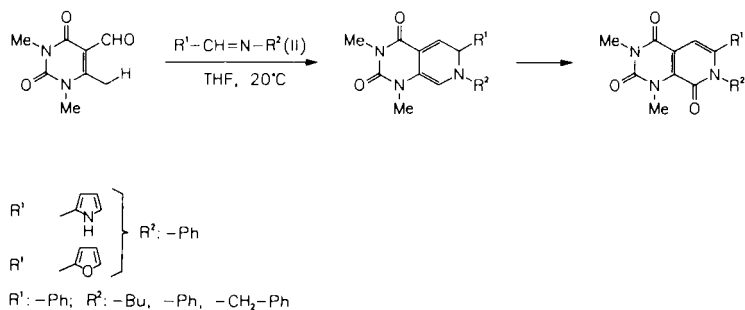
SCHEME 164



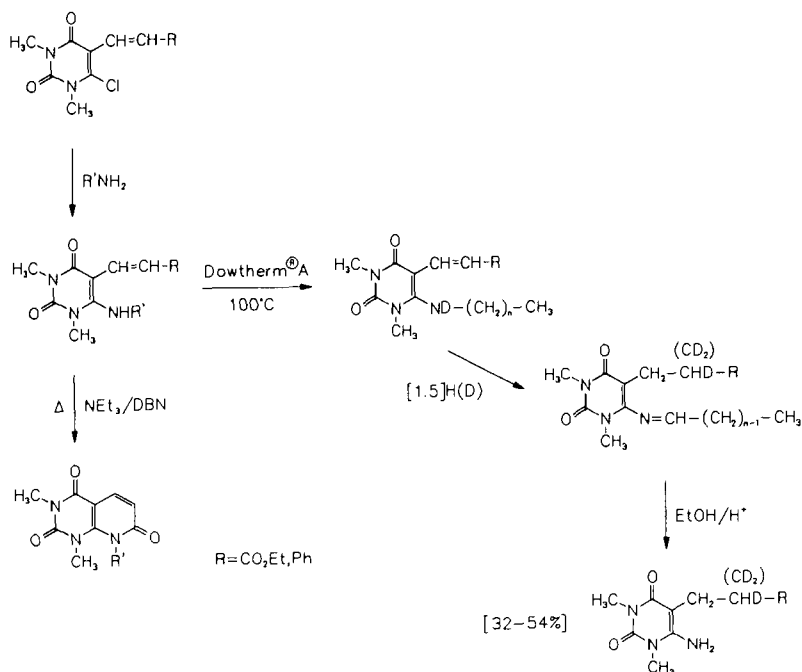
SCHEME 165



SCHEME 166



SCHEME 167



SCHEME 168

XIII. Conclusions

This brief review on uracil and its chemistry, by no means exhaustive, has pointed out by some selective but typical examples that show uracils and their derivatives possess considerable synthetic potential. In most cases, a few easy reaction steps enable the synthesis of novel and highly interesting types of condensed heterocycles which are difficult to obtain by other synthetic means.

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This work is dedicated to the memory of my co-worker and Hungarian friend Géza Szilágyi, an ingenious and brilliant scientist who died too young from a malicious disease.

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Polycyclic Aromatic Nitrogen Cations

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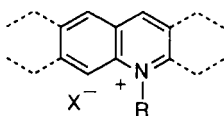
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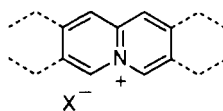
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I. Introduction and Scope

There are two general types of polycyclic aromatic nitrogen cations: *N*-Alkyl quinolinium salts (Type A) and quinolizinium salts (Type B). Since the prefix azonia designates the cationic nitrogen, which is a part of cyclic structures, these two types of compounds are categorized as the azonia derivatives of polycyclic aromatic hydrocarbons. For example, Type A has 1-alkyl-1-azonia naphthalene moiety, and Type B has 4a-azonianaphthalene moiety. Although the chemistry of Type A compounds is an interesting field of aromatic nitrogen cations, this chapter describes only polycyclic aromatic nitrogen cations having a bridgehead nitrogen atom (Type B). In this chapter, the azonia derivatives denote polycyclic aromatic hydrocarbons having bridgehead a nitrogen or nitrogens.



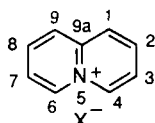
Type A



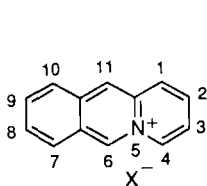
Type B

Since the first synthesis of bicyclic quinolizinium salt (**1**) by Boekelheide and Gall in 1954 (54JA1832), the aromatic cations with a bridgehead nitrogen have attracted much attention because a similar structure is found in alkaloids, such as coralyne, sempervirine, and flavopereirine. Therefore they have been investigated for their biological activities. There has also been considerable interest in the electron-accepting character of these aromatic nitrogen cations. The first systematic review on bridgehead nitrogen heterocycles by Mosby appeared in 1961 (61MI1). It described, in part, polycyclic aromatic nitrogen cations. In work by Thyagarajan [65AHC(5)291], the chemistry of quinolizinium salts has been described. Two comprehensive reviews by Jones [82AHC(31)1] and Bradsher (84MI1) dealt mainly with the chemistry of bicyclic quinolizinium salts

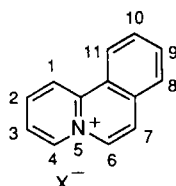
and tricyclic compounds, such as benzo[*b*]quinolizinium (2), benzo[*a*]quinolizinium (3), and benzo[*c*]quinolizinium salts (4). Other reviews also described mainly bicyclic and tricyclic compounds [69ACR181; 75Y GK95; 79MI1; 80H2047; 81H(16)803].



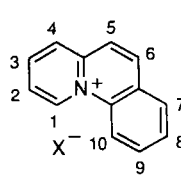
(1)



(2)



(3)



(4)

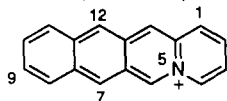
This chapter presents the synthesis of polycyclic aromatic nitrogen cations as well as their chemical and physicochemical properties. The chemistry of bicyclic and tricyclic compounds is also discussed from a theoretical standpoint. Papers published up to the end of 1990 have been covered. Some 1991 references are included.

II. Polycyclic Aromatic Nitrogen Cation Systems

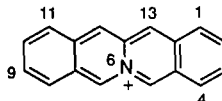
The replacement of a bridgehead carbon of polycyclic aromatic hydrocarbons with nitrogen gives the azonia derivatives. Of the six possible tetracyclic benzenoid aromatic hydrocarbons, one can predict a total of 18 aromatic cations having a bridgehead nitrogen. The parent systems are shown in Table I. The number of mono-azonia derivatives of the 15 possible pentacyclic aromatic hydrocarbons increases to 83, as shown in Table II. In the case of compounds without symmetry, this happens because many isomeric compounds are possible due to the position of quaternary nitrogen. For example, eight isomers are possible in the case of azonia derivatives of benzo[*a*]naphthacene. However, only two compounds (**23** and **24**) have been reported. From these tables, it is apparent that many possible polycyclic aromatic nitrogen cations have not yet been synthesized, and various di- and triazonia compounds are likely target molecules.

TABLE I
TETRACYCLIC BENZENOID AROMATIC NITROGEN CATION SYSTEMS^a

1. Azonia-Naphthacene (2 isomers)

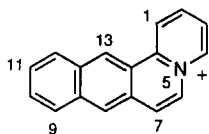


Naphtho[2,3-b]quinolizinium (5)^b

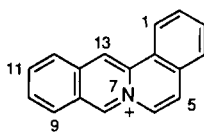


Dibenzo[b,g]quinolizinium (6)

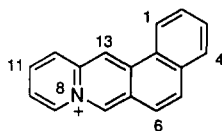
2. Azonia-Benzo[a]anthracene (6 isomers)



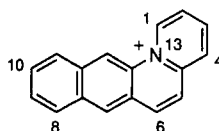
Naphtho[2,3-a]quinolizinium (7)^c



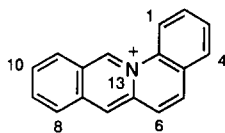
Dibenzo[a,g]quinolizinium (8)



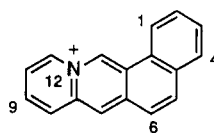
Naphtho[1,2-b]quinolizinium (9)



Naphtho[2,3-c]quinolizinium (10)

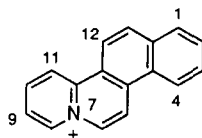


Dibenzo[b,f]quinolizinium (11)^d

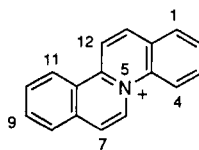


Naphtho[2,1-b]quinolizinium (12)

3. Azonia-Chrysene (3 isomers)

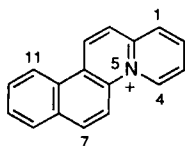


Naphtho[2,1-a]quinolizinium (13)^c



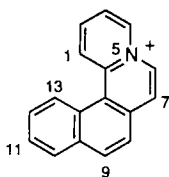
Dibenzo[a,f]quinolizinium (14)

TABLE I (Continued)

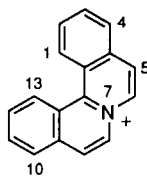


Naphtho[1,2-c]quinolizinium (15)

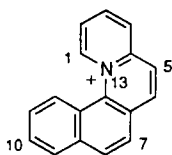
4. Azonia-Benzo[c]phenanthrene (4 isomers)



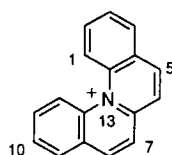
Naphtho[1,2-a]quinolizinium (16)



Dibenzo[a,h]quinolizinium (17)

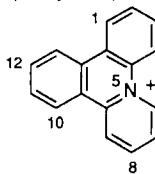


Naphtho[2,1-c]quinolizinium (18)



Dibenzo[c,f]quinolizinium (19)

5. Azonia-Triphenylene (1 isomer)

Pyrido[1,2-f]phenanthridinium (20)^c

6. Azonia-Pyrene (2 isomers)

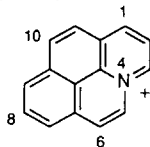
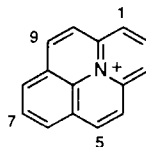
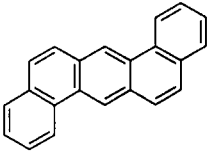
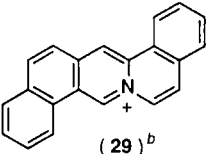
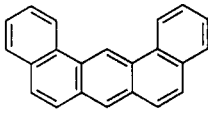
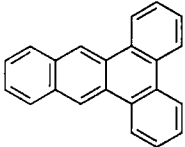
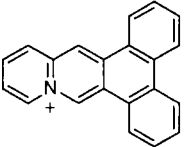
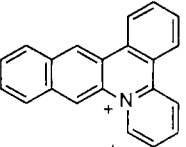
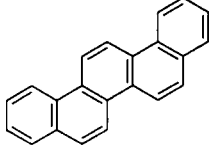
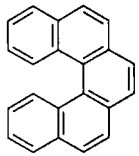
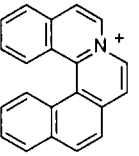
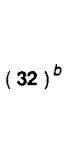
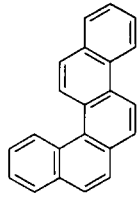
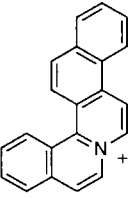
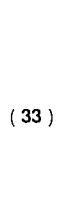
Naphtho[2,1,8-ij]quinolizinium (21)^bQuino[8,1,2-cde]quinolizinium (22)^c^a The nomenclature and numbering follow Chemical Abstracts.^b System has not been synthesized.^c Parent compound has not been synthesized.^d Only UV data has been reported, and the structure has not been completely identified.

TABLE II
AZONIA DERIVATIVES OF PENTACYCLIC BENZENOID AROMATIC NITROGEN CATION SYSTEMS

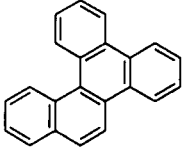
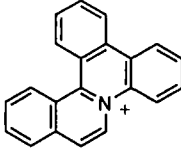
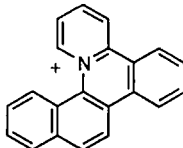
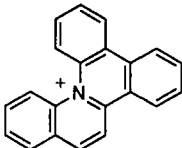
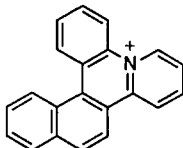
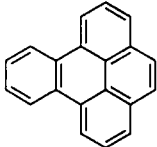
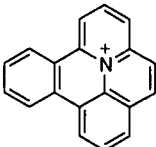
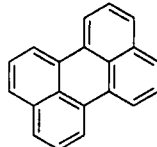
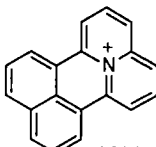
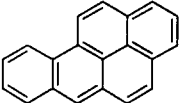
Hydrocarbons	Number of possible monoazonia derivatives	Azonias derivatives reported systems ^a
	4	not synthesized
	8	 (23) (24)
	8	not synthesized
	4	 (25) (26) ^b
	8	 (27) (28) ^b

TABLE II (continued)

	4		
	4	not synthesized	
	4		
	4	not synthesized	
	4		
	8		

(continued)

TABLE II (Continued)

	8		
		(34) ^b	(35) ^b
			
		(36)	(37)
	4		
		(38) ^b	
	3		
		(39)	
	8	not synthesized	

Total 83

^a Chemical Abstracts nomenclatures are shown in text.^b Parent compound has not been reported.

In the following sections, the chemistry of benzenoid and nonbenzenoid polycyclic aromatic nitrogen cations will be described.

III. Theoretical Aspects of Polycyclic Aromatic Nitrogen Cation Systems

A. METHODS OF MOLECULAR ORBITAL TREATMENT

Quantum chemistry is a most useful tool to discuss the structures, physical properties, and reactivities of π -electron systems (61MI2; 69MI1; 81MI1). In quantum mechanics, an electronic state of a molecule is described using a wave function, which is an eigenfunction of a Hamilton operator. Some assumptions must be made to calculate the wave function in the case of a complicated molecule.

1. Assumption 1

A wave function of an electron configuration is generally approximated as a product of molecular orbital functions, which are eigenfunctions of a one-electron Hamilton operator. When $2n$ electrons occupy n molecular orbitals, the wave function of electron configuration of the lowest energy is written as

$$\Phi_1 = \phi_1(1)\alpha(1)\phi_1(2)\beta(2), \dots, \phi_n(2n-1)\alpha(2n-1)\phi_n(2n)\beta(2n) \quad (1)$$

where α and β are spin functions. The full electronic wave function of a ground state is constructed by a linear combination of these $(2n!)$ functions, which is antisymmetric on interchange of any pair of the $2n$ electrons as follows:

$$\Phi = \sum \pm \mathbf{P} \phi_1(1)\alpha(1)\phi_1(2)\beta(2), \dots, \phi_n(2n)\beta(2n) \quad (2)$$

When exchanging operator (\mathbf{P}) corresponds to an even number of exchanges, a positive sign is taken, otherwise a negative sign. The total electronic energy (E) of the singlet ground state of a molecule is calculated using the previously mentioned wave function. If the Hamilton operator (\mathbf{H}) is defined by Eq. (3), the total electronic energy is calculated with the core integrals (I_i) and the electron repulsion integrals (J_{ij} and K_{ij}) [Eq. (4)].

$$\mathbf{H} = \sum_i \mathbf{H}_c + \sum_{i \neq j} \frac{e^2}{r_{ij}} \quad (3)$$

where \mathbf{H}_c denotes the operator of the kinetic energy of an electron and the

potential energy between the cores and electrons, and r_{ij} denotes the distance between electrons i and j .

$$E = \sum_i 2I_i + \sum_{i,j} (2J_{ij} - K_{ij}) \quad (4.1)$$

$$I_i = \int \phi_i(1) \mathbf{H}_c \phi_i(1) d\tau \quad (4.2)$$

$$J_{ij} = \int \phi_i(1) \phi_i(1) \frac{e^2}{r_{12}} \phi_j(2) \phi_j(2) d\tau \quad (4.3)$$

$$K_{ij} = \int \phi_i(1) \phi_j(1) \frac{e^2}{r_{12}} \phi_i(2) \phi_j(2) d\tau \quad (4.4)$$

2. Assumption 2

A molecular orbital is assumed to be represented by a linear combination of atomic orbitals. This assumption is called the linear combination of atomic orbitals (LCAO) approximation. When the atomic orbitals and developing coefficients are denoted by χ_r and C_{rj} , respectively, the molecular orbital (ϕ_j) can be written by

$$\phi_j = \sum_r C_{rj} \chi_r \quad (5)$$

The molecular integrals [Eqs. (4.2)–(4.4)] are calculated by these molecular orbitals of LCAO approximation. In Roothaan molecular orbital theory, the developing coefficients are determined as the electronic energy (E) becomes minimal (51MI1). Then the developing coefficients (C_{rj}) and the molecular orbital energies (ϵ_j) are obtained by solving the Fock equation [Eq. (6.1)].

$$\sum_r C_{rj} (F_{rs} - S_{rs} \epsilon_j) = 0 \quad (6.1)$$

$$F_{rs} = I_{rs} + \sum P_{tu} \{ (rs | tu) - \frac{1}{2} (rt | su) \} \quad (6.2)$$

$$I_{rs} = \int \chi_r(1) \mathbf{H}_c(1) \chi_s(1) d\tau \quad (6.3)$$

$$(rt | su) = \int \chi_r(1) \chi_s(1) \frac{e^2}{r_{12}} \chi_t(2) \chi_u(2) d\tau \quad (6.4)$$

$$S_{rs} = \int \chi_r(1) \chi_s(1) d\tau \quad (6.5)$$

P_{ii} is the electron density (q_i) at the core (i), and P_{iu} is the bond order (p_{iu}) between cores i and u .

3. Assumption 3

If differential overlaps $[\chi_r(1) \chi_s(1) d\tau]$ are assumed to be nearly equal to zero, Eq. (6.2) can be rewritten as

$$F_{rr} = I_{rr} + \frac{1}{2} q_r(rr | rr) + \sum'_s q_s(rr | ss) \quad (6.6)$$

$$F_{rs} = I_{rs} - \frac{1}{2} p_{rs}(rr | ss) \quad (6.7)$$

The core integrals (I_{rr}) are approximated by Eq. (7.1), using the valence state ionization potentials [$I_p(r)$] of the core (r),

$$I_{rr} = -I_p(r) - \sum'_{s \neq r} n_s (rr | ss) \quad (7.1)$$

where n_s denotes the charge of the core (s). The second term of Eq. (7.1) approximately corresponds to the sum of the potential energies by the core (s), other than core (r). Then, Eq. (6.6) can be rewritten as

$$F_{rr} = -I_p + \frac{1}{2} q_r(rr | rr) + \sum'_s (q_s - n_s) (rr | ss) \quad (7.2)$$

When all electron densities are near to the corresponding core charges, the core integrals F_{rr} can be approximated as

$$F_{rr} = -I_p + \frac{1}{2} n_r(rr | rr) \quad (7.3)$$

4. Assumption 4

Equation (7.3) tells us that the coulomb integrals ($\alpha_r = F_{rr}$) are determined only by the character of the core (r). If bond orders are not so largely different from each other, the resonance integrals ($\beta_{rs} = F_{rs}$) are also determined by the character of the bond between the cores r and s .

When the π -electron system consists of carbon atoms and heteroatoms, which are not so largely electronegative, the following set of approximations is frequently used.

- (1) The coulomb integrals (α_r) are written as $\alpha_r = \alpha + h_r \beta$, where α and β are the coulomb integral of the carbon atom and the resonance integral of the carbon-carbon bond, respectively. They are assumed to depend only on the character of a carbon atom.
- (2) The resonance integrals β_{rs} are written as $\beta_{rs} = k_{rs} \beta$. The value of k_{rs} is assumed to be nonzero only when atoms r and s are linked to each other and to depend proportionally on the overlap integrals.
- (3) The overlap integrals between the different cores are neglected in Eq. (6.1).

These approximations are called the Hückel approximation. The molecular orbital theory using LCAO and Hückel approximations is called the Hückel molecular orbital theory (HMO). HMO gives nearly correct and reasonable results when the distribution of the charge density does not largely deviate from homogeneity. However, HMO calculations cannot

give adequate results in the case of π -electron systems containing more electronegative heteroatoms.

5. Assumption 5

If the electron repulsion $[rr | ss (r \neq s)]$ is ignored, the coulomb integrals ($\alpha_r = F_{rr}$) can be written as

$$\alpha_r = \alpha_r^0 + \omega(n_r - q_r)\beta \quad (7.4)$$

where

$$\alpha_r^0 = -I_p + \frac{1}{2} n_r(rr | rr),$$

and the electron repulsion integrals $(rr | rr)$ are taken to be $2\omega | \beta |$. The value of ω is assumed to be constant for all of the core. The molecular orbital method using Eq. (7.4) is developed by Streitwieser and is called the omega method. The omega method can lead to improved results within the framework of the HMO (61MI2).

More explicit molecular orbital methods including the electronic repulsion terms are called advanced molecular orbital methods. In such methods, F matrix elements of the Fock equation are calculated by Eqs. (6.6) and (6.7), and the iterative procedure, or self-consistent field (SCF) procedure must be used. The most convenient advanced molecular orbital method for the polycyclic aromatic compounds is the semiempirical method suggested by Pariser, Parr, and Pople (the PPP method). In this PPP method, the required integral values are empirically determined using the following approximations.

6. Assumption 6

The resonance integrals (I_{rs}) are calculated by the overlap integrals. The electron repulsion integral of type $(rr | rr)$ is approximately determined using the ionization potential $[I_p(r)]$ and the electron affinity $[A(r)]$ of each core.

$$(rr | rr) = I_p(r) - A(r) \quad (8)$$

The electron repulsion integrals $(rr | ss)$ between different cores r and s are generally calculated by the Nishimoto-Mataga equation.

7. Assumption 7

The wave function of the electronic system of a planar molecule can be separated into the π and σ parts. The σ part is assumed to form the core

framework field of the molecule, which the π electrons move over. This assumption is called the π approximation.

The following discussions are mainly based on the π approximation.

B. ELECTRONIC STRUCTURES OF AROMATIC NITROGEN SYSTEMS

Heteroaromatic compounds are isoconjugate with the corresponding aromatic hydrocarbons (the parent hydrocarbons). For example, the parent of quinolizinium cation (**1**) is naphthalene. The difference in the electronic energies between the heteroaromatic compound and the parent hydrocarbon can be expressed by first order perturbation theory as follows:

$$\Delta E = \sum_r q_r (\alpha_N - \alpha) \quad (9)$$

where q_r is the electron density at the core (r). Since $(\alpha_N - \alpha)$ is always negative, the π -electron energy of the heteroaromatic compound is lower than that of the parent hydrocarbon.

Delocalization energy or resonance energy is defined as the difference between the formation energy of the aromatic compound and the summation of bond energies of the double and single bonds constituting the molecule. When the parent hydrocarbon is an alternant hydrocarbon, which does not have an odd-membered ring, all the π -electron densities are in unity (Coulson–Rushbrook’s theorem). Then, in this case, ΔE nearly equals the product of the number of heteroatoms and $(\alpha_N - \alpha)$. Similarly, the difference in the summation of the bond energies between the heteroaromatic compound and the parent hydrocarbon also nearly equals the product of the number of heteroatoms and $(\alpha_N - \alpha)$. Therefore, the delocalization energy of the heteroaromatic compound is nearly that of its alternant parent hydrocarbon in the first-order approximation. Table III shows that the calculated values support the previous considerations (8IM11).

On the other hand, when the parent hydrocarbon is a nonalternant hydrocarbon, having an odd-membered ring, the delocalization energy of the heteroaromatic compound is lower than that of the parent, because Coulson–Rushbrook’s theorem cannot be valid.

An alternant hydrocarbon is a bichromatic system in which the cores are classified into two parities, starred and unstarred, and no cores of like parity are directly linked. Perturbation theory suggests that a heteroatom gathers the electron density from the carbon atoms of the opposite parity, as shown by the electron densities of alternant aromatic nitrogen cation

TABLE III
 RESONANCE AND DELOCALIZATION ENERGIES

Compounds	RE(kcal mol ⁻¹)		DE ^c /β
	I ^a	II ^b	
Hydrocarbons:			
Benzene	35.9	20.0	2.00
Naphthalene	61.0	30.5	3.68
Anthracene	83.5	36.9	5.32
Phenanthrene	91.3	44.6	5.45
Heteroaromatics:			
Pyridine	27.9	20.9	1.87
Quinoline	48.4	33.0	3.58
Isoquinoline	—	34.1	3.56
Acridine	—	41.3	5.24
Pyrrole	21.6	8.5	—
Furan	16.2	1.6	—
1	—	—	3.89
2	—	—	5.53
3	—	—	5.66
4	—	—	5.67

^a Vertical resonance energy (49CB358).

^b Dewar resonance energy, which is well known to be the most reasonable definition of resonance energy (69MII).

^c Delocalization energy calculated by the omega method (M. Hida, unpublished data).

systems obtained by different molecular orbital (MO) calculations (Table IV).

A polycyclic aromatic molecule having an odd-membered ring is an anion or mesoionic electronic system. Distributions of electron density calculated by the PPP method are compared in Table V. Negative charge mainly distributes over the five-membered ring in indene anion (**47**). Electron in indolizine (**43**) is gathered on the bridgehead nitrogen atom, but the electron densities distribute approximately uniformly over the other cores of the molecule.

All of the molecular orbital energies are lowered by substitution of a

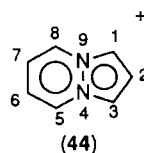
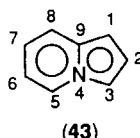
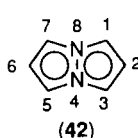
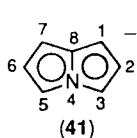
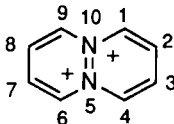


TABLE IV
ELECTRON DENSITIES OF AROMATIC NITROGEN CATION SYSTEMS

Quinolinizinium (1):														
Position	1	2	3	4	5	9a							Refs.	
HMO ^b	1.005	0.916	1.011	0.856	1.550	0.874							<i>a</i>	
omega	0.968	0.932	0.973	0.896	1.564	0.898							<i>a</i>	
PPP ^c	1.010	0.976	1.013	0.940	1.185	0.940							81RTC161	
PPP ^c	0.939	0.912	0.889	1.065	1.340	1.047							68TCA(11)417	
Benzo[<i>b</i>]quinolinizinium (2):														
Position	1	2	3	4	5	6	7	8	9	10	11	11a	Refs.	
HMO ^b	1.004	0.939	1.013	0.877	1.552	0.791	0.968	1.000	0.968	1.005	1.013	0.905	<i>a</i>	
omega	0.972	0.950	0.978	0.913	1.567	0.865	0.977	0.984	0.973	0.988	0.969	0.922	<i>a</i>	
PPP ^c	0.931	0.950	0.875	1.106	1.333	1.044	0.996	0.908	0.922	0.978	0.897	1.094	70G421	
Benzo[<i>a</i>]quinolinizinium (3):														
Position	1	2	3	4	5	6	7	8	9	10	11		Refs.	
HMO ^b	1.006	0.895	1.011	0.833	1.555	0.901	1.019	1.006	0.977	1.000	0.979		<i>a</i>	
omega	0.967	0.919	0.971	0.882	1.568	0.930	0.983	0.992	0.980	0.988	0.982		<i>a</i>	
PPP ^c	0.953	0.905	0.916	1.040	1.338	1.139	0.848	0.952	0.930	0.921	0.982		70G421	
Pyridazo[1,2- <i>a</i>]pyridazinium (40):														
Position	1	2	3	4	5	10							Refs.	
HMO ^b	0.738	0.909	0.909	0.738	1.707	1.707							<i>a</i>	
omega	0.830	0.902	0.902	0.830	1.537	1.537							<i>a</i>	
PPP ^c	0.978	0.810	0.810	0.978	1.423	1.423							68TCA(11)417	

^a M. Hida, unpublished results.^b HMO, Hückel molecular orbital theory.^c PPP, Pariser-Parr-Pople method.

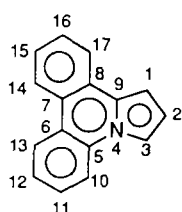
hetero atom for a carbon atom. This suggests that the polycyclic aromatic compounds containing a nitrogen atom possess larger electron accepting character, but less electron donating character than the parent hydrocarbons. Therefore, their properties and reactivities will differ considerably from those of the parent hydrocarbons as mentioned in Section V,C and VI.

C. HYBRIDIZATION OF BRIDGEHEAD NITROGEN ATOMS

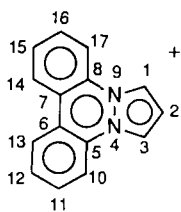
The semiempirical PPP method assuming the π approximation has been widely used with a great deal of success in studying the electronic struc-

TABLE V
ELECTRON DENSITIES OF AROMATIC COMPOUNDS HAVING AN ODD-MEMBERED RING

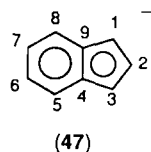
Positions	1	2	3	4	5	6	7	8	9	Refs.
41	1.270	1.088	1.391	1.387	1.391	1.088	1.270	1.114		68TCA(11)411
42	1.281	0.997	1.281	1.440	1.281	0.997	1.281	1.440		68TCA(11)411
43	1.134	1.016	1.238	1.387	1.120	0.981	1.049	0.949	1.124	69T2259
44	1.058	0.974	1.058	1.476	1.064	0.913	0.913	1.064	1.476	69T2259
47	1.216	1.115	1.216	1.080	1.043	1.104	1.104	1.043	1.080	91UPI
Positions	1	2	3	4	5	6	7	8	9	Refs.
45	1.087	1.054	1.180	1.453	1.066	1.008	1.037	0.982	1.143	69T2259
46	1.021	1.021	1.021	1.489	1.149	1.012	1.012	1.149	1.489	69T2259
Positions	10	11	12	13	14	15	16	17		
45	1.025	1.987	0.996	0.988	0.993	1.010	0.988	1.002		
46	0.989	0.943	0.923	0.963	0.963	0.923	0.943	0.989		



(45)



(46)



(47)

tures of π -electron systems. However, the assumption of π electrons moving in the potential field of a framework of σ bonds may be a rather crude approximation in the case of nitrogen containing heteroaromatic systems, because the presence of nitrogens in a crucial position in the polycyclic systems might evoke considerable polarization of the σ -core, which could break down the π approximation.

One rather satisfactory way is to introduce all valence electrons in the molecular orbital approach without a hypothesis on the hybridization of atomic orbitals. Population analysis using the CNDO/2-SCF-MO showed the electronic configuration of the nitrogen atom of aromatic nitrogen cation systems (**1**, **40**, **41**, **42**, **43**, **44**) in the ground state to be as follows (69G1078):

- (**1**): $1s^2 2s^{1.19} 2p\sigma^{2.51} 2p\pi^{1.31}$
 (**40**): $1s^2 2s^{1.21} 2p\sigma^{2.36} 2p\pi^{1.28}$
 (**41**): $1s^2 2s^{1.19} 2p\sigma^{2.42} 2p\pi^{1.47}$
 (**42**): $1s^2 2s^{1.21} 2p\sigma^{2.26} 2p\pi^{1.42}$
 (**43**): $1s^2 2s^{1.19} 2p\sigma^{2.46} 2p\pi^{1.40}$
 (**44**): $1s^2 2s^{1.20} 2p\sigma^{2.29} 2p\pi^{1.43}$

The hybridization on the nitrogen atom is $s^a p\sigma^b$ with $a = 1.20$ and $b = 2.36 - 2.51$, which is dramatically different from the $s^1 p\sigma^2$ trigonal hybrid. The discrepancy is more marked in the case of compounds **1**, **40**, **41**, and **43** with one bridgehead nitrogen atom. On the other hand, the hybridization on carbon atoms is close to the $s^1 p\sigma^2$ trigonal hybrid.

The π population carried by the $2p\pi$ atomic orbital at the bridgehead nitrogen decreases progressively as the number of bonds forming the fused bicyclic n/m ring systems increases. As shown in Table VI, a similar tendency is observed in the case of the parent hydrocarbons. Table VI

also shows that the difference in π -electron density at the bridgehead position between the heteroring system and the corresponding hydrocarbon increases as the ring size (n/m) increases. These results are very reasonable, because the nitrogen atom can attract electrons more easily from the other part of the molecule as the ring size increases.

IV. Syntheses

A. BENZENOID AROMATIC NITROGEN CATIONS

1. Bicyclic Aromatic Nitrogen Cations (Quinolizinium Salts)

The synthesis of the bicyclic quinolizinium system has been reviewed by Thyagarajan [65AHC(5)291], Hida (75YKG95), Jones [82AHC(31)1], and Bradsher (84MI1). Hence only the syntheses of the parent quinolizinium salt (**1**) and its monosubstituted derivatives will be highlighted.

The unsubstituted quinolizinium salt (**1**) was first synthesized by Boekelheide and Gall (54JA1832). The reaction of 2-picolylolithium with β -ethoxypropionaldehyde gave alcohol **48** (57% yield), which was treated with hydroiodic acid and then alkali to give cyclic alcohol **49** (61%). Dehydration with acetic anhydride quantitatively afforded dihydroquinolizinium

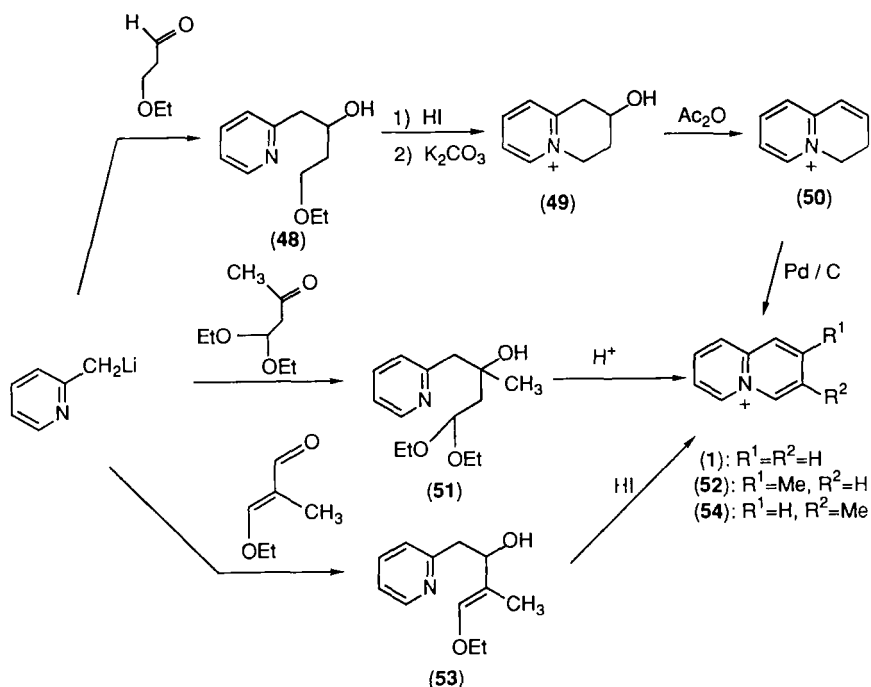
TABLE VI
RING SIZE AND π POPULATION AT BRIDGEHEAD CORE

Compound	ring size (n/m)	π Population	
		CNDO/2 ^a	PPP ^b
41	(5/5)	1.469	1.387 (1.142)
43	(5/6)	1.404	1.387 (1.080)
1	(6/6)	1.309	1.340 (1.000)
42	(5/5)	1.425	1.440 (1.142)
44	(5/6)	1.430	1.476 (1.080)
40	(6/6)	1.278	1.423 (1.000)

^a Calculated by complete neglect of differential overlap (CNDO/2) (69G1078).

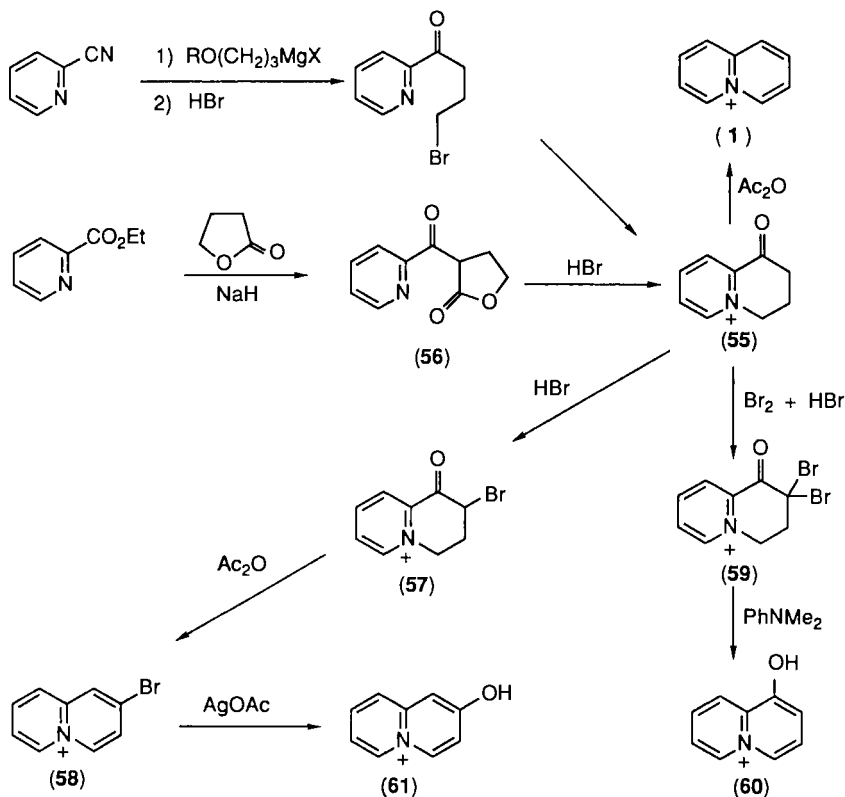
^b Calculated by PPP method (M. Hida, unpublished results). The values at the bridgehead carbon atom of the corresponding hydrocarbons are compared in parentheses.

ion **50**, which was dehydrogenated with Pd/C to yield **1** in low yield. The reaction of 2-picolyllithium with 4,4-diethoxybutan-2-one or 2-methyl-3-ethoxyacrolein gave alcohol **51** or **53**, which cyclized under acidic conditions to 2-methyl- or 3-methylquinolizinium salt (**52** or **54**), respectively (58JCS3067; 86CB2062).

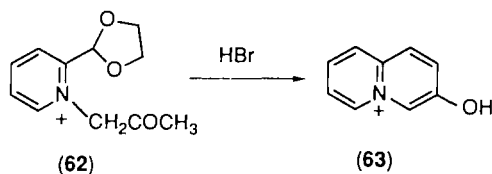


Glover and Jones found that cyclic ketone **55** was aromatized by refluxing in acetic anhydride to afford **1** (96%) (58JCS3021). Ketone **55** was obtained in two different ways. One is the reaction of 2-cyanopyridine with a Grignard reagent followed by treatment with HBr [56CI(L)1456]. By using 2-acetylpyridine and a Grignard reagent from 3-chloropropionaldehyde diethyl acetal as starting compounds, 1-methylquinolizinium salt was obtained (59JCS1686). The other method for the synthesis of ketone **55** was reported by Miyadera and Iwai (64CPB1338). The condensation of

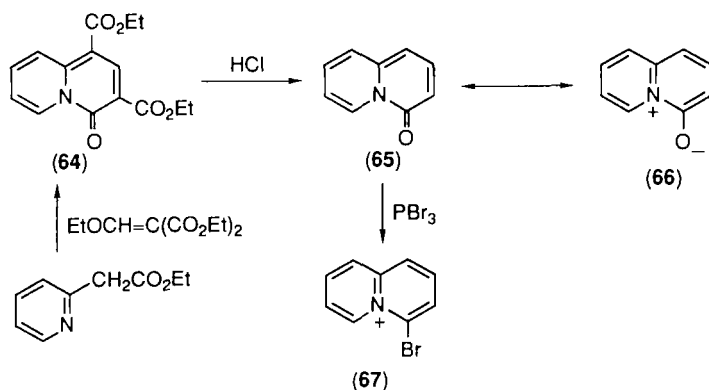
2-ethoxycarbonylpyridine with γ -butyrolactone in the presence of sodium hydride yielded keto lactone **56** (69.5%), which was cyclized with hydrobromic acid to afford **55** (82.5%). By using this method, four (mono-methyl)quinolizinium salts were prepared.



When cyclic ketone **55** was reacted with hydrobromic acid, bromoketone **57** was obtained (89%). The Glover and Jones aromatization of **57** afforded 2-bromoquinolizinium salt (**58**: 74%) (63JCS2203). Bromination of **55** under drastic conditions gave dibromoketone **59** (91%), which was converted to 1-hydroxyquinolizinium salt (**60**: 78%) by refluxing with dimethylaniline (63JCS2203). 2-Hydroxyquinolizinium salt (**61**) was prepared by treatment of **58** with silver acetate in refluxing acetic acid (90%) (64JCS2760). 3-Hydroxy derivative **63** was obtained by intramolecular condensation of 1-acetyl-2-(2-dioxolanyl)pyridinium bromide (**62**) in 50% hydrobromic acid (96%) (65JOC526).



Ethyl 2-pyridylacetate reacts with diethyl ethoxymethylene malonate to give cyclic compound **64**, which, on refluxing in hydrochloric acid, affords 4-quinolizone (**65**) [51JA3681]. The 40% contribution of the betaine form **66** to the resonance hybrid was estimated on the basis of the ^1H -NMR spectrum of **65** [73JOC4391].

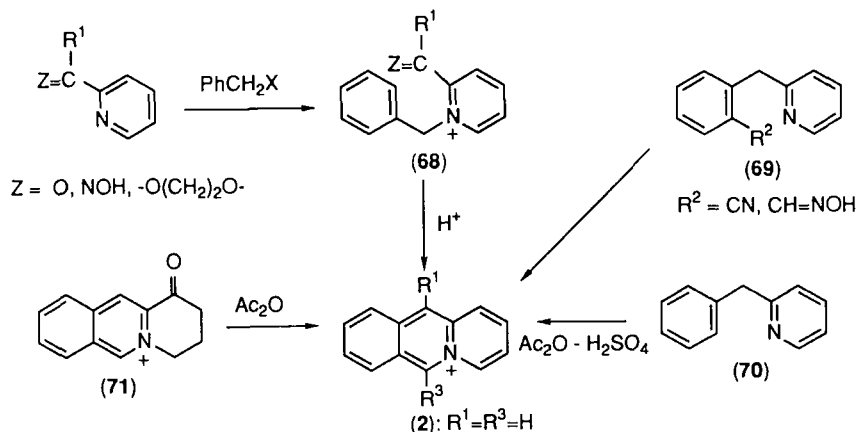


The reaction of **65** with phosphorus tribromide gave 4-bromo derivative **67** (45%) [81H(15)213]. 1-Bromoquinolizinium salt was synthesized by heating quinolizinium perbromide at 200°C (69%) [81H(15)213]. Sanders and co-workers used Miyadera's method to prepare a 3-bromo derivative. 5-Bromo-2-ethoxycarbonylpyridine and γ -butyrolactone were used as starting compounds [81H(15)213].

2. Tricyclic Aromatic Nitrogen Cations

Typical examples of tricyclic aromatic nitrogen cations are summarized for comparison with the synthesis of polycyclic aromatic nitrogen cations, since the reviews by Bradsher (69ACR181; 84MI1) and Jones [82AHC(31)1] have described synthetic methods for tricyclic ring systems. Recent developments also will be described.

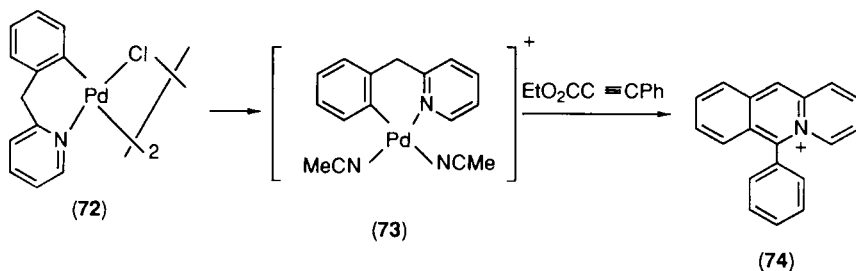
a. *Benzo[b]quinolizinium Salts*. The most general synthetic method for producing the benzo[*b*]quinolizinium ring is the cyclodehydration of 1-benzylpyridinium salts under acidic conditions. In the first report by Bradsher and Beavers (55JA4812), quaternary salt **68** obtained by the reaction of 2-pyridinecarbaldehyde with benzyl bromide, which was heated with 48% HBr to afford parent compound **2** (60%). Bradsher and co-workers modified this approach and developed the synthesis of many benzo[*b*]quinolizinium derivatives (69ACR181; 84MI1). Thus, instead of the aldehyde, a reaction with the oxime and the acetal gave good results. Cyclization also occurs in the presence of PPA, HCl, HF, and H₂SO₄. In the case of 2-cyanopyridine, cyclization with sulfuric acid at 100°C gave 11-amino derivative (35%) (73JOC4167).



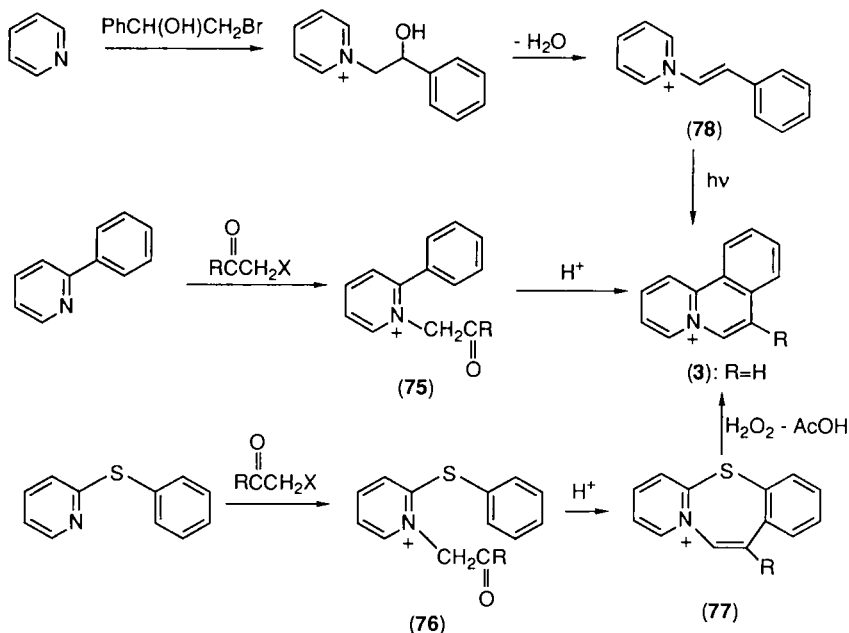
Alternative routes to this system starting from 2-benzylpyridine derivatives **69** have been reported (67JOC733; 71JHC157; 78JOC3536; 80JOC4248). Cyclization of 2-benzylpyridine derivatives **70** with acetic anhydride-sulfuric acid was also useful for the preparation of 6-methyl derivatives (60JOC293). Glover and Jones (58JCS3021) reported the treatment of cyclic ketone **71** with boiling acetic anhydride to afford **2** (62%).

A new method using an organopalladium compound was reported by Maasarani and Pfeffer (90MI1). Cationic compound **73** derived from chloro-bridged dimer **72** reacted with 3-phenylpropionate in chlorobenzene to give 6-phenylbenzo[*b*]quinolizinium salt **74** (17%).

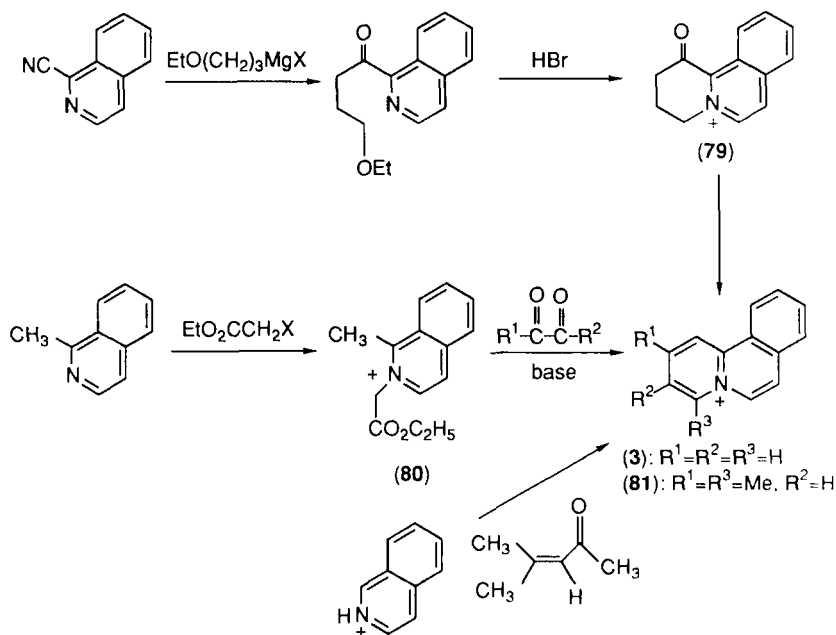
b. *Benzo[a]quinolizinium Salts*. Three routes starting from pyridine derivatives have been reported. The cyclodehydration method by Bradsher and Beavers (55JA453) is useful for substituted benzo[*a*]quinoli-



zinium salts. Quaternary salts **75** derived from 2-phenylpyridines with α -halo ketones were heated with HBr to give 7-substituted benzo[*a*]quinolinizinium salts. With Bradsher's sulfur extrusion method (62JOC4478), the salt **76** prepared from 2-phenylthiopyridine and α -halo ketone was cyclized to afford thiazepinium salt **77**. Sulfur extrusion was accomplished on treatment with hydrogen peroxide in acetic acid to afford **3**. Although these two methods gave the 7-substituted **3** inevitably, the use of the oxime of chloroacetaldehyde instead of α -halo ketones affords parent compound **3** (63JOC3205; 66JOC978). The photocyclization of 1-styryl pyridinium salts **78** also is a convenient method for constructing this ring system (66JOC2616).

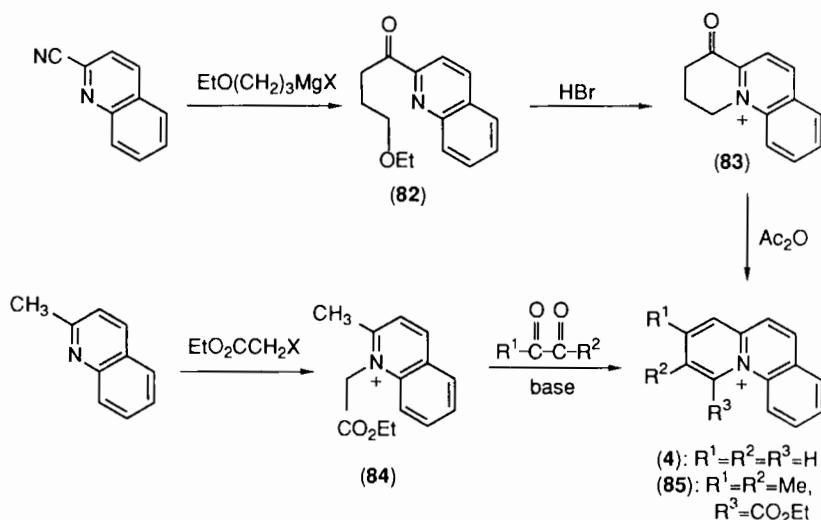


There are three methods using isoquinoline derivatives as starting compounds. Glover and Jones aromatization of cyclic ketone **79** with boiling acetic anhydride gave **3** (66%) (58JCS3021). 2,3,4-Trisubstituted compounds were obtained by the Westphal condensation of 1-methyl-2-ethoxycarbonylmethylisoquinolinium salt (**80**) with α -diketone in the presence of anhydrous sodium acetate (86JHC1151). 2,4-Dimethylbenzo[*a*]quinolizinium salt (**81**) was prepared by the reaction of isoquinolinium perchlorate with mesityl oxide at 120°C in 35% yield.



c. *Benzo[*c*]quinolizinium Salts.* There are five methods to construct benzo[*c*]quinolizinium salts. The first synthesis of parent compound **4** was accomplished by Glover and Jones (58JCS3021). The reaction of 2-cyanoquinoline with Grignard reagent gave ketone **82**, which was treated with HBr to afford cyclization product **83** (75%). The cyclic ketone **83** was converted to **4** in boiling acetic anhydride (63%). The Westphal condensation was applied to the synthesis of this tricycle (61AP37). The reaction of 1-ethoxycarbonylmethyl-2-methylquinolinium salt (**84**) with α -diketone proceeded in water using sodium bicarbonate base to afford 1-ethoxycarbonyl-2,3-dimethyl derivative **85** (28%) (86JHC1151).

By using the intramolecular thermal cyclization reported by Fozard and Bradsher, substituted **4** can be obtained (66JOC2346). The *cis*-2-[2-(2-

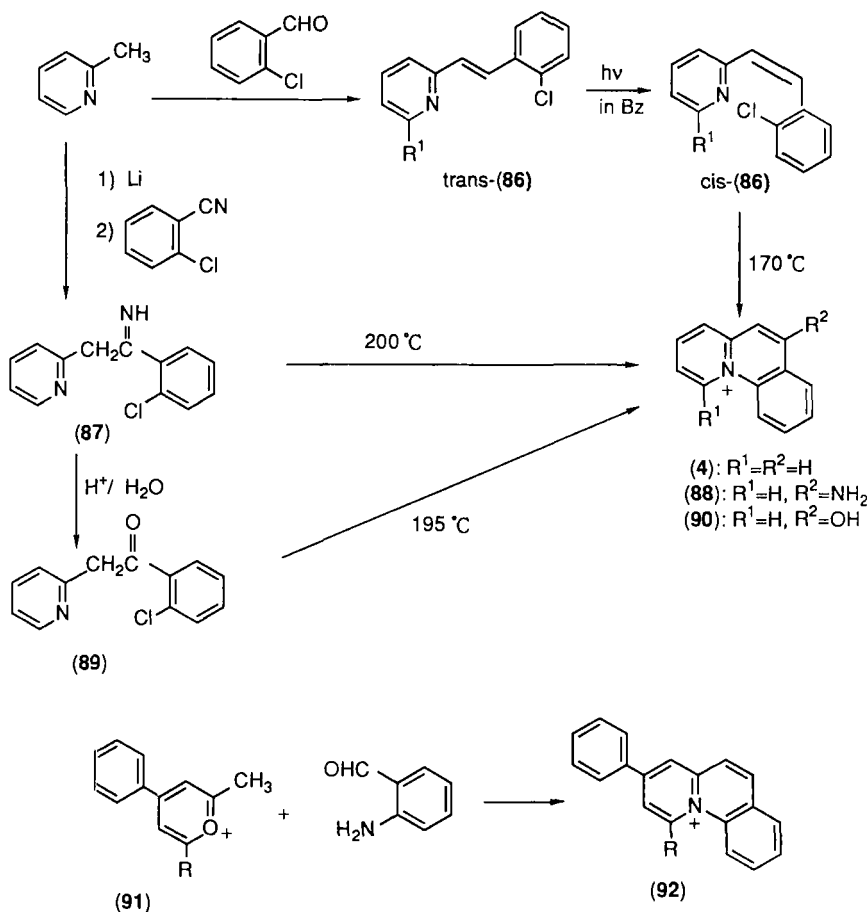


chlorophenyl]vinyl]pyridine (*cis*-**86**; $\text{R} = \text{H}$) obtained by photoisomerization of the *trans* form in benzene was heated at 170°C to give **4** (50%). The introduction of a nitro group in the benzene ring increased the ease of cyclization: 4-methyl-8-nitro derivative (80%) was obtained on cyclization at 25°C for 48 hours, whereas 4-methyl derivative (69%) needed the reaction at 165°C for 1 hour. However, the cyclization to the 1-methyl derivative failed. Arai *et al.* reported that the 1-methyl derivative (56%) was obtained by irradiating an acetonitrile solution of *trans*-**86** ($\text{R}^1 = \text{CH}_3$) (91CL1355). In this photocyclization, it is essential to select the solvent (acetonitrile) and irradiation wavelength ($290 < \lambda < 340 \text{ nm}$ and $\lambda > 400 \text{ nm}$).

The 6-amino **88** (58%) and 6-hydroxy **90** (84%) derivatives were synthesized by the cyclization of imine **87** and ketone **89**, respectively (79JHC753). 1,3-Disubstituted derivatives **92** were prepared by the reaction of pyrylium salts **91** with *o*-aminobenzaldehyde (71% for **92**; $\text{R} = \text{Me}$) (71TL553).

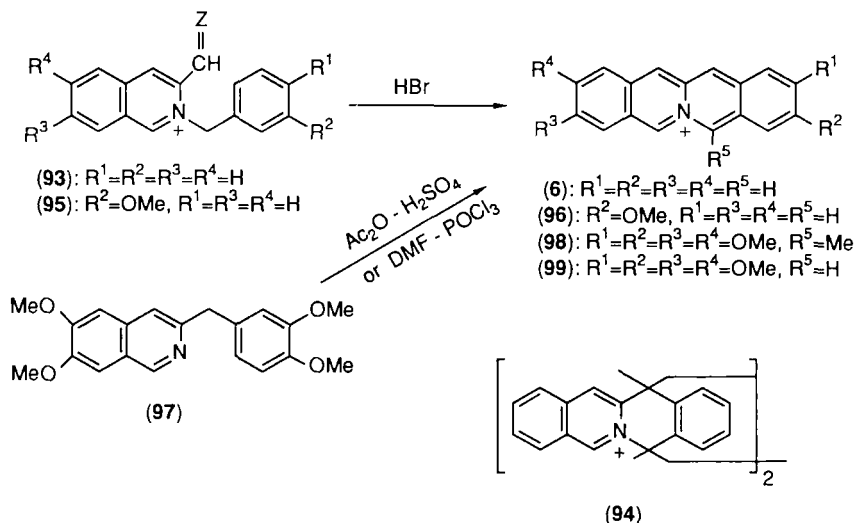
3. Tetracyclic Aromatic Nitrogen Cations

a. *Dibenzo[b,g]quinolizinium Salts*. The cyclodehydration method for the synthesis of **2** was applied to linearly condensed tetracycle **6** (60JA1808). The cyclization of salt **93** ($\text{Z} = \text{O}$) with HBr for 8 hours, however, afforded nonfluorescent dimer **94** of **6**. The unsubstituted **6** (23%) was obtained together with dimer **94** (35%) by treating **93** ($\text{Z} = \text{NOH}$) with

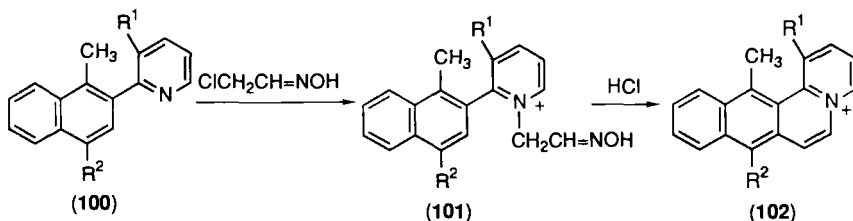


boiling HBr for 10 min. Salt **6** was dimerized under conditions with boiling HBr or with light. By introducing a methoxy group para to the cyclization position (**95**), salt **96** was obtained in good yield (78%) (60JOC191).

5-Methyl-2,3,9,10-tetramethoxy derivative (stracoralayne) **98** which has a yellow fluorescence, is the linear analogue of alkaloid coralyne (8-methyl-2,3,10,11-tetramethoxydibenzo[*a,g*]quinolinizinium salt **110**). It was obtained by treating 3-benzylisoquinoline derivative **97** with $Ac_2O-H_2SO_4$ in 48% yield (68AP33). The treatment of **97** with $POCl_3$ -dimethylformamide (DMF) (Vilsmeier conditions) gave tetramethoxy derivative **99** [70ZN(B)1408]. Cheng observed that the aqueous solution of salt **98** was unstable, and the UV spectra changed on standing overnight (76JMC882).

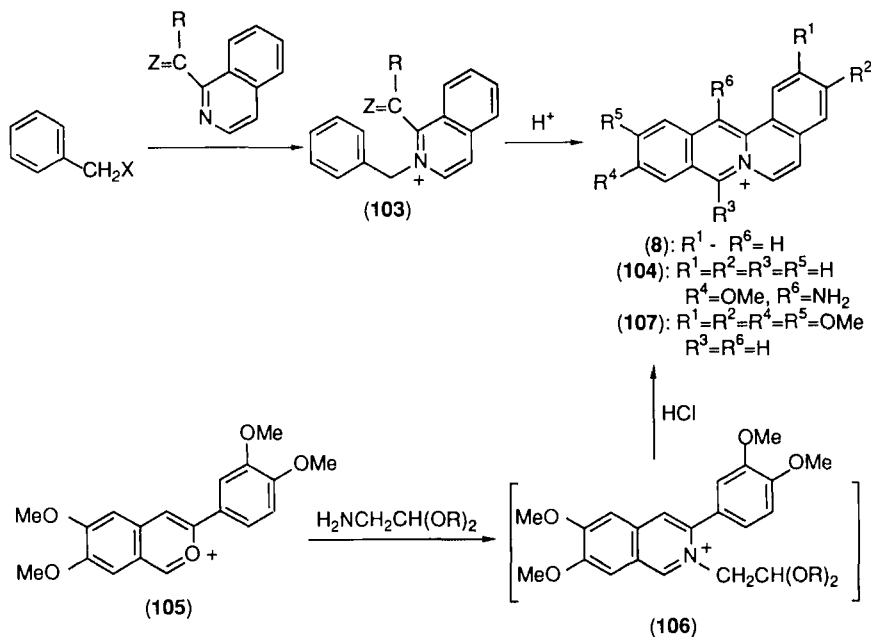


b. *Naphtho[2,3-a]quinolizinium Salts*. The only example of the synthesis of this tetracycle utilized the cyclodehydration reaction (65JHC399). 1-Methyl-2-(2-pyridyl)naphthalene derivatives **100**, derived from 2-acetonylpyridines in three steps, were quaternized by chloroacetaldoxime in 72–90% yields. Salts **101** were cyclized under acidic conditions to afford 13-methyl derivatives **102** (76% for $\text{R}^1 = \text{R}^2 = \text{H}$; 82% for $\text{R}^1 = \text{H}, \text{R}^2 = \text{CH}_3$; 48% for $\text{R}^1 = \text{R}^2 = \text{CH}_3$).



c. *Dibenzofa,g]quinolizinium Salts*. Many papers have been written on the synthesis of this system because it corresponds to the alkaloid coralyne structure. Protoberberine alkaloids also have a partially reduced form. These derivatives are highly fluorescent and show antileukemic activity. The synthetic methods are categorized into three reactions: cyclodehydration, coralyne reaction, and photocyclization.

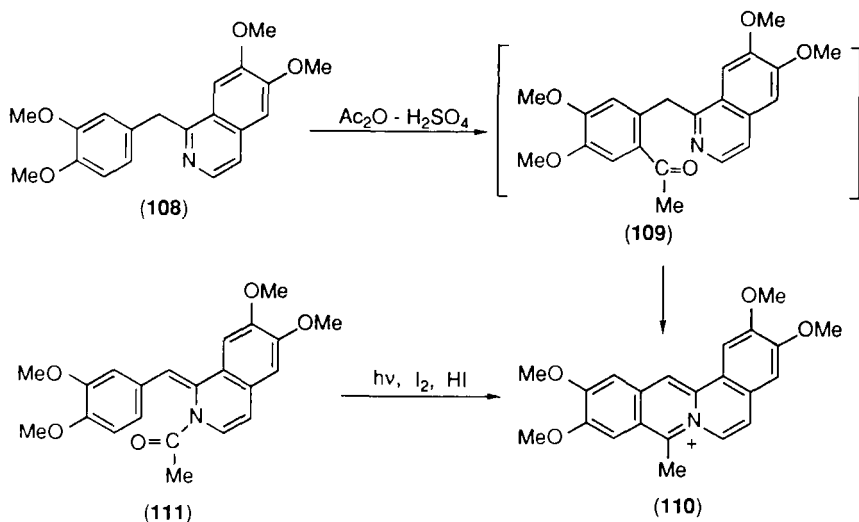
Since Bradsher obtained parent compound **8** (52%) by cyclodehydration of the quaternary salt **103** ($R = H, Z = O$) derived from 1-isoquinolinecarbaldehyde and benzyl bromide (58JOC430), many derivatives have been synthesized by this method. The oxime (60JOC757) and the acetal (65JOC752) were also used instead of the aldehyde. 13-Amino-10-methoxy derivative **104** (88%) was obtained by cyclodehydration of the salt derived from 1-cyanoisoquinoline and 3-methoxybenzyl bromide (73JOC4167). A Russian group reported the synthesis of tetramethoxy derivative **107** by the cyclodehydration of 3-arylisquinolinium salt **106**, obtained from benzo[*c*]pyrylium salt **105** (89KPS75).



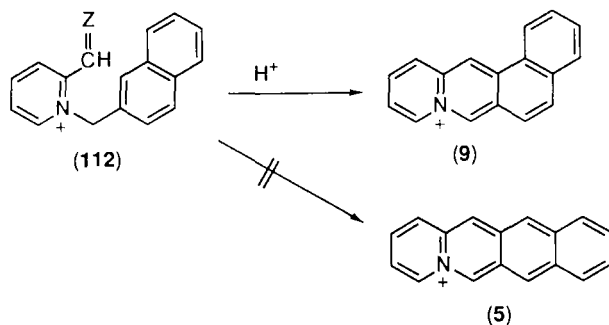
The cyclization of 1-benzylisoquinoline derivatives under acidic conditions is also useful for synthesizing this system. In a synthesis reported by Schneider and Schroeter (20CB1459), papaverine (**108**) was treated with acetic anhydride and sulfuric acid to give coralyne (**110**). By using this coralyne synthesis or a Vilsmeier–Haack reaction with a mixture of POCl_3 and DMF, 1-benzylisoquinoline derivatives were converted to dibenzo[*a,g*]quinolizinium salts [70ZN(B)1408; 74YZ478; 76JMC882]. Under these reactions acetyl **109** or a formyl intermediate was considered to cyclize under acidic conditions.

The only example of a photocyclization appeared in a Japanese patent

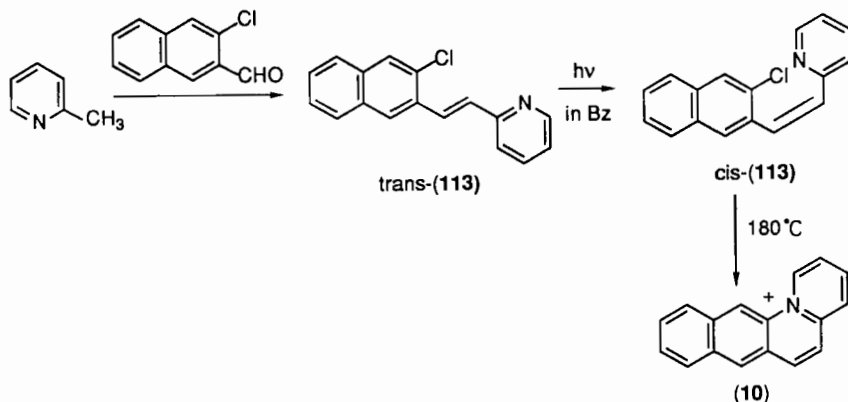
[76JAP(K)76/34200]: Irradiating a methanol-tetrahydrofuran (THF) solution of amide **111** in the presence of iodine and HI afforded coralyne. The chemical properties of coralyne and related compounds have been examined [75JPS1825; 80JCS(P1)911, 80JCS(P1)919; 81JHC223].



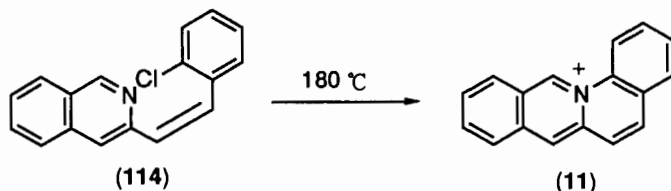
d. *Naphtho[1,2-b]quinolizinium Salts*. Salt **112** ($\text{Z} = \text{O}$), obtained by the reaction of 2-bromomethylnaphthalene and 2-pyridinecarbaldehyde, was cyclized with HBr at position 1 of the naphthalene ring to afford parent compound **9** (72%) (56JA2459). On the basis of its UV spectra, the angular structure **9** rather than the linear structure **5** was proposed. PPP calculations support this result (see Table IX). The acetal or the oxime instead of the aldehyde was used for the synthesis of substituted **9** (64JHC121; 84JHC261).



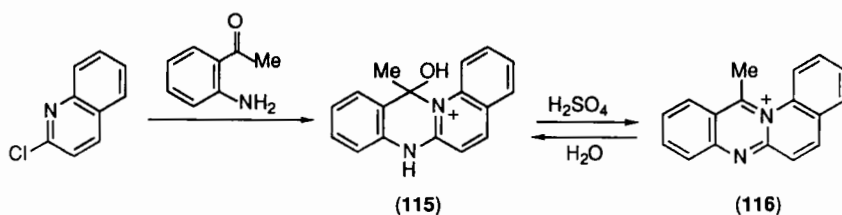
e. *Naphtho[2,3-c]quinolizinium Salts*. The condensation of 2-methylpyridine with 3-chloro-2-naphthalenecarbaldehyde in acetic anhydride gave *trans*-**113**, which was isomerized in benzene by irradiation with Pyrex-filtered light to the *cis*-**113**. The intramolecular thermal cyclization of *cis*-**113** at 180°C afforded **10** in 27% yield (66JOC3683).



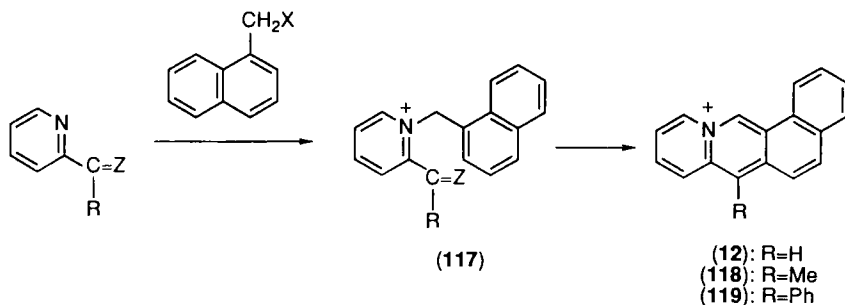
f. *Dibenzo[b,f]quinolizinium Salts*. Fozard and Bradsher reported that on heating *cis*-3-styrylisoquinoline **114**, cyclization product **11** was obtained in low yield (66JOC3683). Although they could not confirmed the structure, the reported UV data is nearly identical with that obtained by PPP calculation on structure **11** (see Table IX).



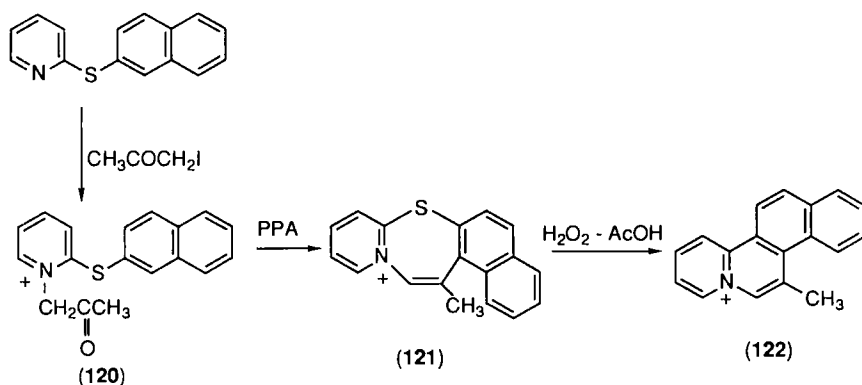
The reaction of 2'-aminoacetophenone with 2-chloroquinoline gave tetracycle **115** (87%). The pseudo-base **115** was dehydrated with sulfuric acid to yield aza derivative **116** of salt **11** (65JOC1539). The structural assignment of **116** was based on ¹H-NMR and UV spectral data. Further work seems needed.



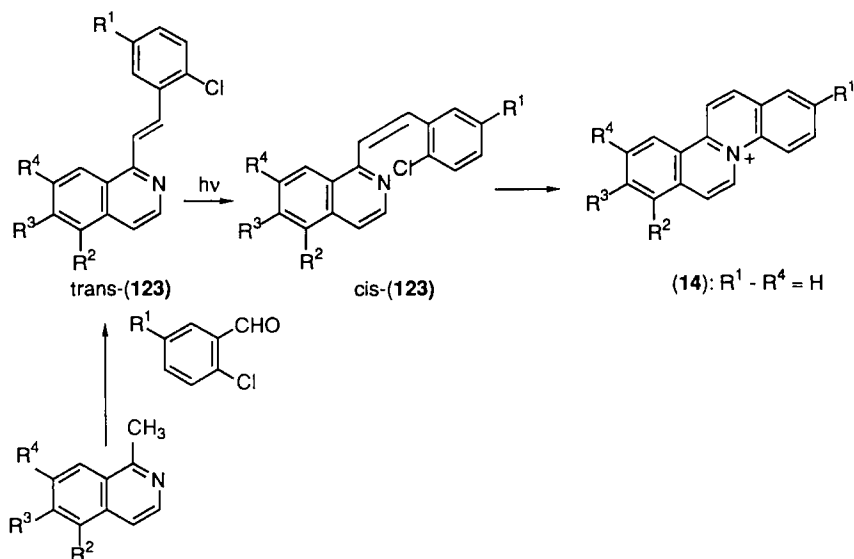
g. *Naphtho[2,1-b]quinolizinium Salts*. Bradsher and co-workers used the cyclodehydration of salt **117** ($R = H$, $Z = O$) for the synthesis of **12**. The yield was increased from 52 to 78% by the use of the oxime (60JOC757) instead of 2-pyridinecarbaldehyde (56JA2459). By using 2-acetylpyridine and 2-phenacylpyridine as starting compounds, **118** and **119** were obtained, respectively (59JOC589).



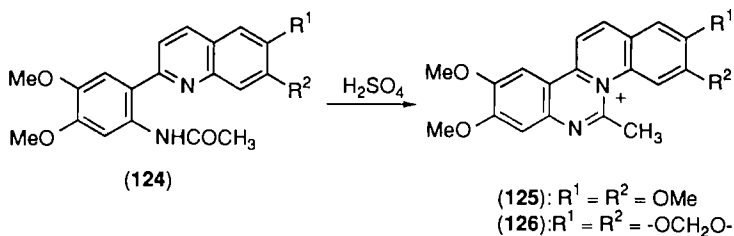
h. *Naphtho[2,1-a]quinolizinium Salts*. Quaternary salt **120** (88%), obtained by the reaction of 2-(2-naphthyl)thiopyridine with iodoacetone, was cyclized with polyphosphoric acid (PPA). The resulting thiazepinium salt **121** was treated with hydrogen peroxide in acetic acid to afford 5-methyl derivative **122** of **13** (37%) (62JOC4482).



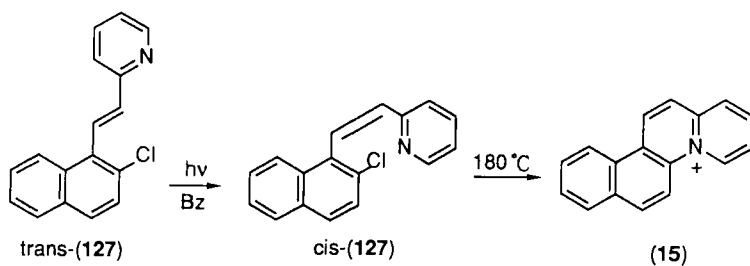
i. *Dibenzof[a,f]quinolizinium Salts*. The thermal cyclization of *cis*-1-styrylisoquinoline **123** ($R = H$) at 200°C afforded parent compound **14** (70%) (66JOC3683). In the case of *trans*-stilbazole **123** ($R^1 = NO_2$) with a nitro group para to a chloro group, the irradiation of the benzene solution directly afforded the derivatives of **14** (85% for $R^1 = NO_2$, $R^2 = H$, $R^3 = R^4 = OMe$; 80% for $R^1 = NO_2$, $R^2 = R^3 = OMe$, $R^4 = H$) (70JHC1421).



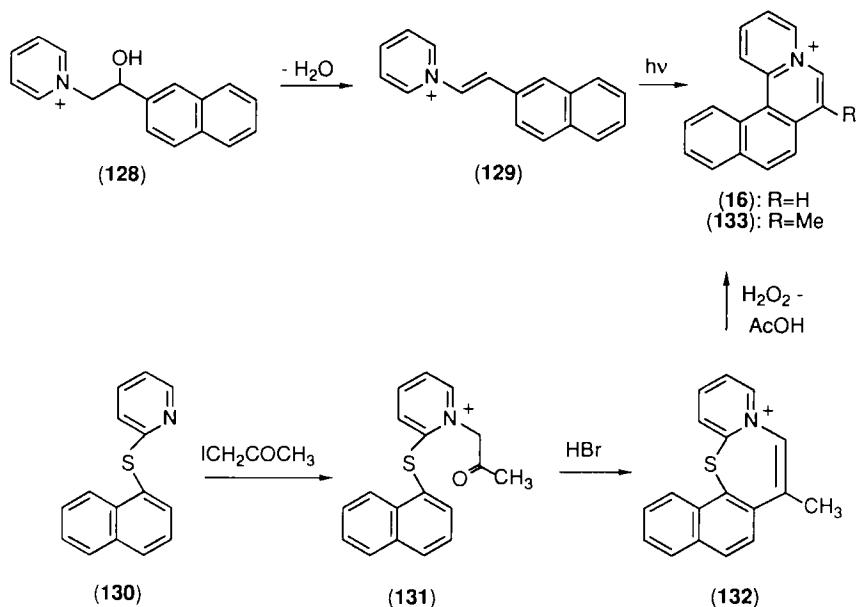
Phillips and Castle reported the synthesis of aza derivatives **125** (72%) and **126** (10%) by the cyclization of 2-(2-acetamidophenyl)quinoline derivatives **124** with conc. H_2SO_4 (80JHC1489).



j. *Naphtho[1,2-c]quinolizinium Salt.* The thermal cyclization of *cis*-**127** at $180^\circ C$ gave parent compound **15** (75%) (66JOC3683).

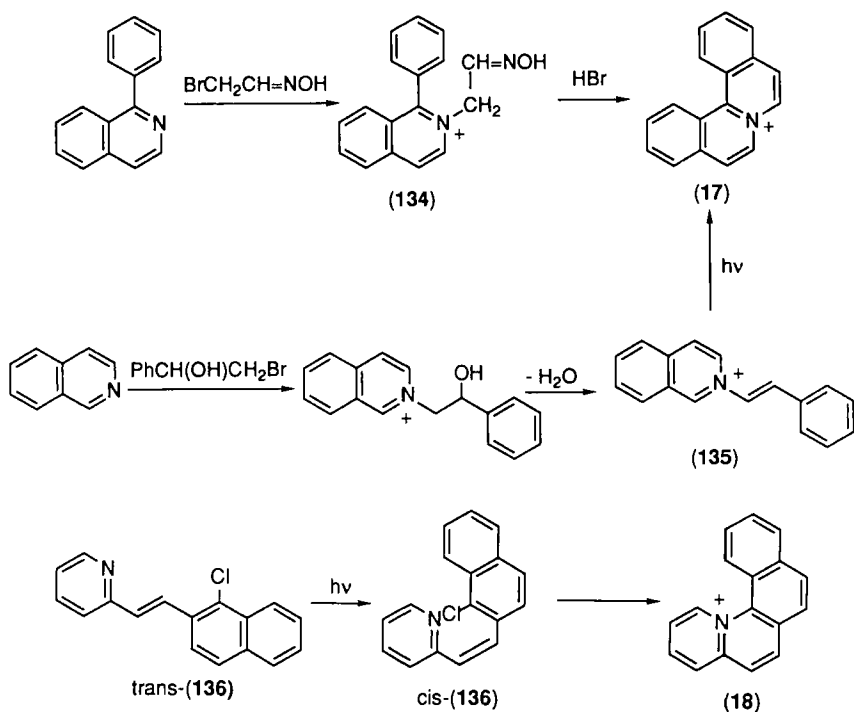


k. *Naphtho[1,2-a]quinolizinium Salts*. Two syntheses have been reported for this system. Arai and Hida used photocyclization to synthesize parent compound **16**. The dehydroxylation of alcohol **128** afforded 1-[2-(2-naphthyl)vinyl]pyridinium salt (**129**). Irradiation with Pyrex-filtered light of a methanolic solution of olefin **129** afforded compound **16** (45%) [87JCS(P1)481]. The sulfur extrusion reaction was applied to prepare the methyl derivative **133** (62JOC4482). The sulfide **130** was quaternized with iodoacetone to give salt **131** (85%), which was cyclized under acidic conditions to afford thiazepinium salt **132** (73%). Treatment of **132** with hydrogen peroxide afforded **133** (60%).



l. *Dibenzo[a,h]quinolizinium Salts*. Parent compound **17** was obtained by the cyclodehydration (74%) of salt **134** formed by the reaction between 1-phenylisoquinoline and 2-bromoacetaldoxime (65JOC1846) or by the photocyclization of 2-styrylisoquinolinium salt **135** in methanol (24%) [87JCS(P1)481].

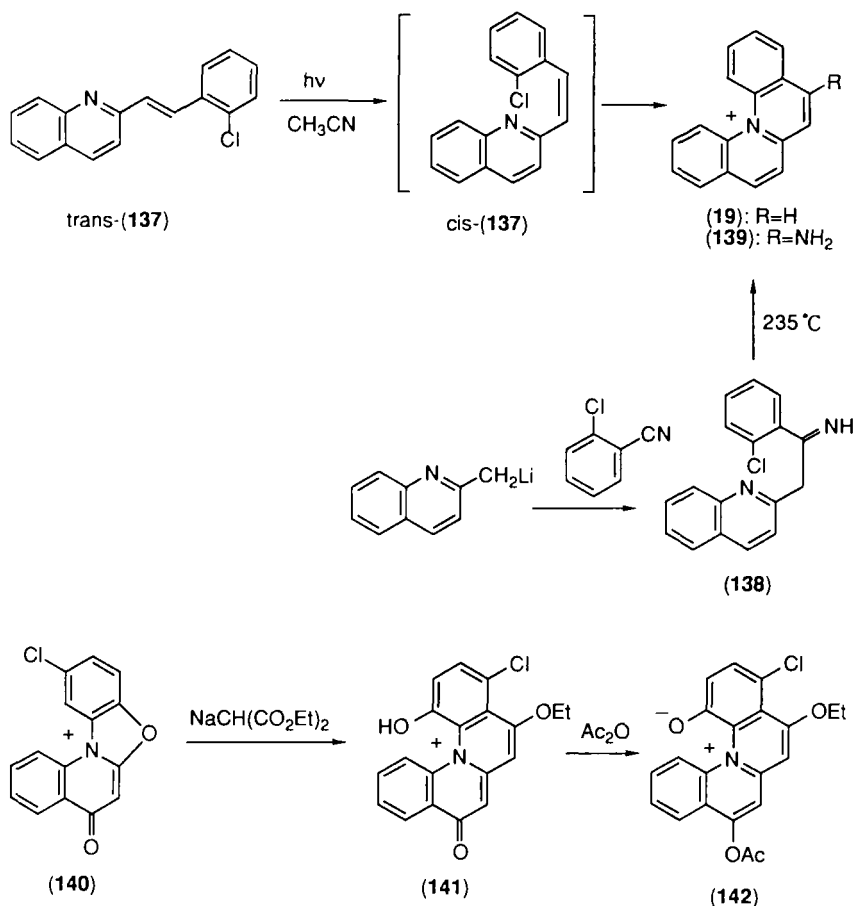
m. *Naphtho[2,1-c]quinolizinium Salts*. Only one example depicting the synthesis of **18** is a thermal cyclization. Thus, the condensation of 2-methylpyridine with 1-chloro-2-formylnaphthalene gave *trans*-**136**. The *trans*-**136** was isomerized with UV light to *cis*-**136**, which was cyclized at 180°C to give **18** (50%) (66JOC3683).



n. *Dibenzo[c,f]quinolinizinium Salts*. Although the thermal cyclization of *cis*-**137** was unsuccessful, parent **19** was obtained by irradiating an acetonitrile solution of *trans*-**137** using selected wavelength light ($280 < \lambda < 360$ nm and $\lambda > 430$ nm) (91CL1355).

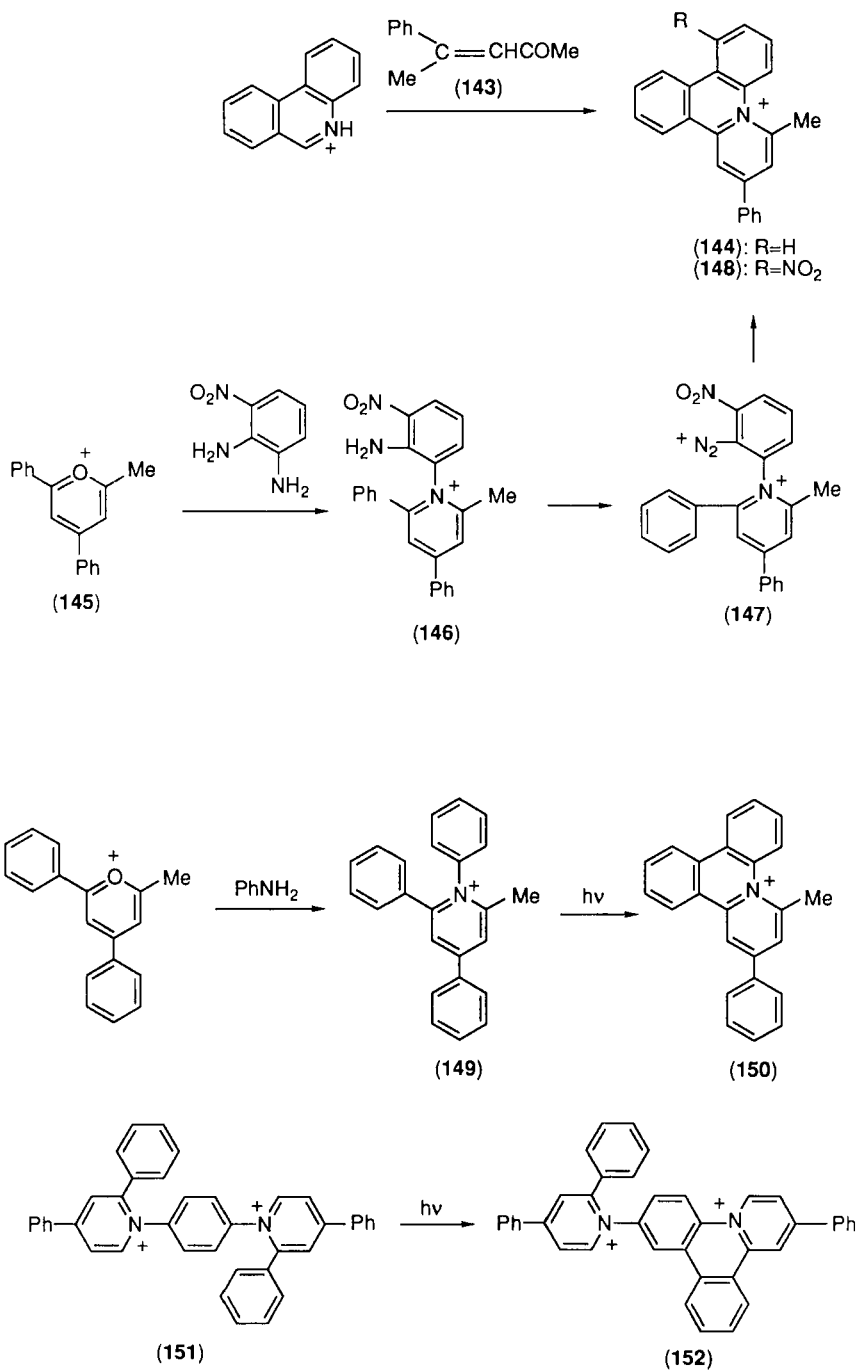
Vierfond *et al.* used thermal cyclization (79JHC753). The anion obtained by the reaction of quinaldine with phenyllithium was reacted with *o*-chlorobenzonitrile to afford imine **138**, which cyclized at 235°C to give amino derivative **139** (32%). Another method is shown by the reaction of **140**–**142**. The reaction of **140** with excess sodium diethyl malonate in hexamethylphosphoramide (HMPA) at 190°C gave cyclization product **141** (38%), which was converted to fully aromatic **142** by reaction with acetic anhydride (92%) (82JHC127).

o. *Pyrido[1,2-f]phenanthridinium Salts*. Three types of reactions are available for synthesizing this system. Chapman reported a one-step reaction between phenanthridinium perchlorate with unsaturated ketone **143** to afford 6-methyl-8-phenyl derivative **144** (75CC489). A Russian group reported the synthesis of **148** by a Pshorr reaction. The reaction of pyrylium salt **145** with *o*-phenylene diamine gave amino substituted 1-phenyl-

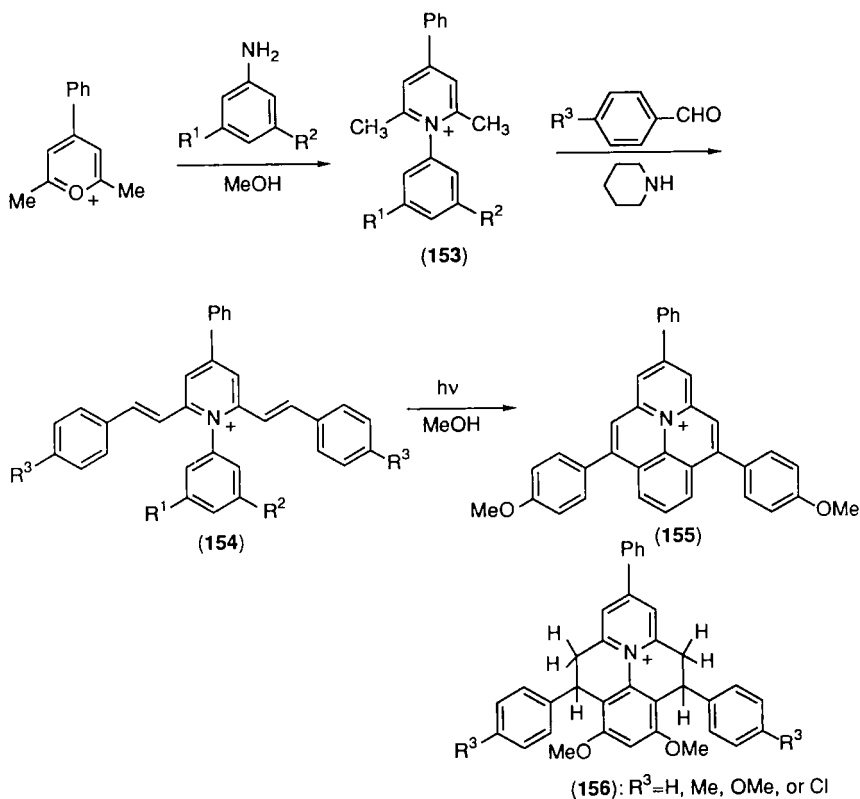


pyridinium salt **146** (97%), which was treated with nitrosyl perchlorate to give diazonium salt **147**. Salt **147** was cyclized on treatment with copper powder to yield 1-nitro derivative **148** (77%) (74KGS1344; 78KGS1226). Hydrogenation with Pd/C gave the 1-amino derivative quantitatively. Acylation and diazotization of the amino group did not proceed, presumably because of the low basicity.

The Katritzky group and a Russian group published independently the synthesis of tetracyclic system **150** by photocyclization. Irradiating a methanolic solution of 2-methyl-1,4,6-triphenylpyridinium salt (**149**) gave 6-methyl-8-phenyl derivative **150** [76ZOR1126; 79CC268; 80JCS(P1)1879; 82IZV535; 84KGS1509; 88DOK1435]. By using this method, many derivatives were prepared. In the case of irradiation of the bispyridinium salt **151**, only monocyclization product **152** was obtained (80MI1; 85ZOR1136).

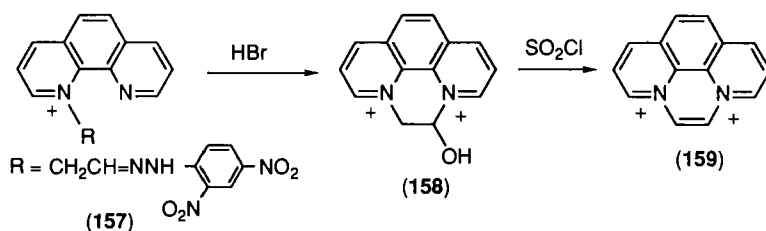


p. *Quino[8,1,2-cde]quinolizinium Salts*. The only synthetic example of this system was photocyclization reported by Soroka (89CS361). Methyl substituted 1-arylpyridinium salt **153** reacted with benzaldehyde derivatives to afford distyryl derivatives **154**. The methanolic solution of **154** was irradiated with a mercury lamp. The distyryl derivative **154** with a *p*-methoxy group in the styryl moiety ($R^1 = R^2 = \text{H}$, $R^3 = \text{OMe}$) afforded azoniapyrene derivative **155** in 0.1% yield. Other styryl derivatives with a 1-(3,5-dimethoxyphenyl)pyridinium moiety gave tetrahydropyrene derivatives **156** in 60–77% yields.



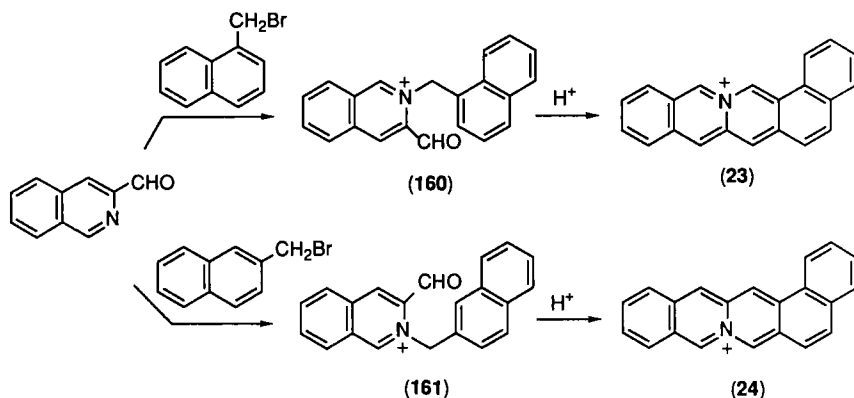
q. *Pyrazino[1,2,3,4-lmn]-1,10-phenanthrolinium Salts*. Black and Summers synthesized the diazonia derivative **159** of pyrene (68T6453), which is related to bipyridinium herbicides. The reaction of 1,10-phenanthroline with bromoacetaldehyde 2,4-dinitrophenylhydrazone in benzene gave quaternary salt **157** (92%). Salt **157** was cyclized on treatment with conc. HBr to afford alcohol **158** (70%), which was dehydrated with thionyl

chloride to give **159** (45%). This compound, however, was inactive as a herbicide.



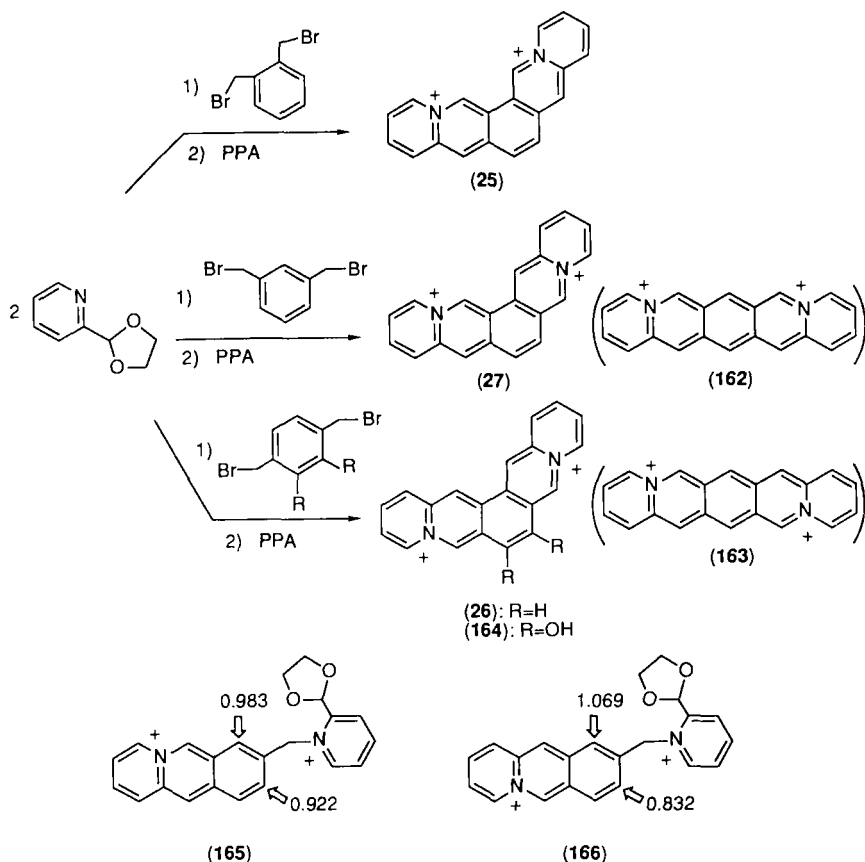
4. Pentacyclic Aromatic Nitrogen Cations

a. *Azoniabenzo[a]naphthacenes*. The cyclodehydration of salts **160** and **161** with HBr gave benzo[*b*]naphtho[1,2-*g*]quinolizinium (**23**) (43%) and benzo[*b*]naphtho[2,1-*g*]quinolizinium salts (**24**) (66%), respectively (60JOC191).

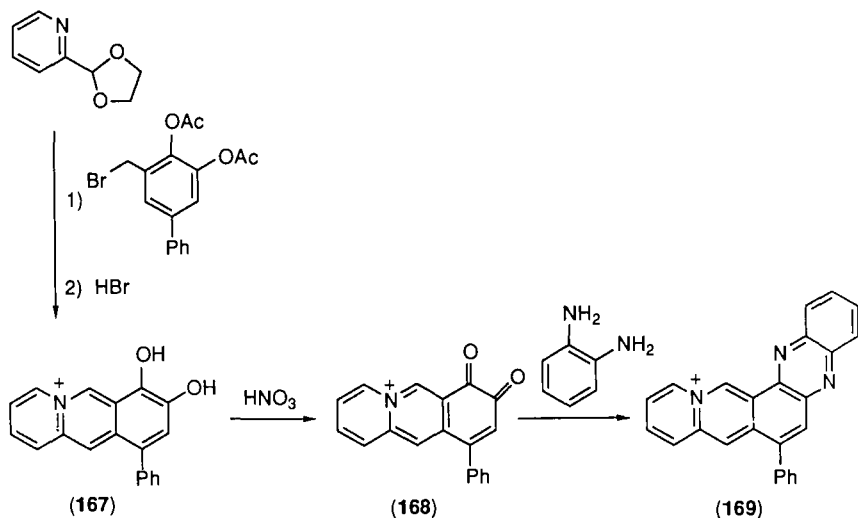


b. *Azoniapentaphenes*. Diazonia derivatives of pentaphene have been synthesized by cyclodehydration (64JOC856). The salts obtained from the reaction of 2-(1,3-dioxolan-2-yl)pyridine and 1,2-di(bromomethyl)benzene were heated at 150–160°C in PPA to afford dipyrido[1,2-*b*:2',1'-*j*][2,9]phenanthrolinediium salt (**25**) (48%). The products from salts with 1,3- and 1,4-di(bromomethyl)benzene were not linear benzo[1,2-*b*:5,4-*b'*]diquinolizinediium salt (**162**) and benzo[1,2-*b*:4,5-*b'*]diquinolizinediium salt (**163**), but dipyrido[1,2-*b*:1',2'-*j*][2,8]phenanthrolinediium salt (**27**) (54%) and dipyrido[2,1-*b*:1',2'-*j*][3,8]phenanthrolinediium salt (**26**) (65%), respectively. The angular cyclization is supported by MO calculations.

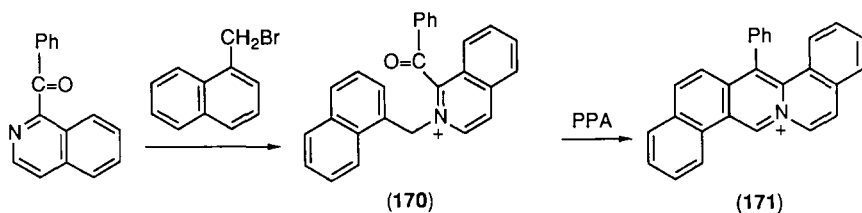
Cyclization of intermediates **165** and **166** occurred at the position with a larger superdelocalizability (see Section VI,A). PPP Calculations also support the angular forms. These compounds were not stable; their color changed even in polar solvents. The salt from the reaction of 3,6-bis(bromomethyl)catechol diacetate with 2-(1,3-dioxolan-2-yl)pyridine was easily cyclized with 30% HBr in AcOH to afford the 6,7-dihydroxy derivative **164** (84%) (65JOC252).



Fields and Miller reported a pentacyclic system incorporating quinoxaline. 7,8-Dihydroxybenzo[*b*]quinolizinium salt (**167**), obtained by cyclodehydration (92%), was oxidized with nitric acid to yield *o*-quinone **168** (84%). Azoniaanthraquinone **168** was easily condensed with *o*-phenylenediamine to afford 7-phenylquinolizino[3,2-*a*]phenazin-13-ium salt (**169**) with a yellow fluorescence (89%) (70JHC91).

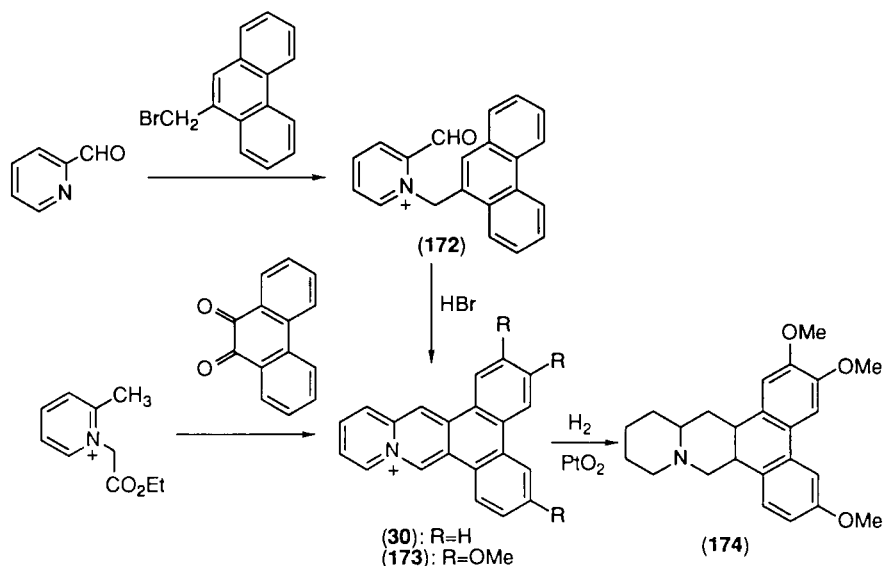


c. *Azoniadibenzof[a,h]anthracene*. Salt **170** derived from 1-benzoyl-isoquinoline and 1-bromomethylnaphthalene was cyclized with PPA at 140–150°C to yield 15-phenylbenzo[*a*]naphtho[1,2-*g*]quinolizinium salt (**171**) (56%) (59JOC589).

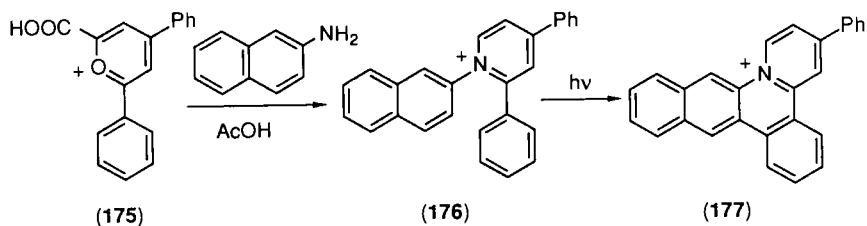


d. *Azoniadibenzof[a,c]anthracenes*. Two isomeric systems (**30** and **31**) have been reported. The reduced form of **30** is related to the alkaloid cryptopleurine. The synthesis of **30** has been accomplished either by cyclodehydration or the Westphal condensation. Bradsher used cyclodehydration to synthesize the parent compound phenanthro[9,10-*b*]quinolizinium salt (**30**). Salt **172** obtained from 9-bromomethylphenanthrene and 2-pyridine carbaldehyde, was treated with hydrobromic acid to afford cyclization product **30** (24.5%) (56JA2459). The trimethoxy derivative **173** was also obtained by this method and was hydrogenated to give (\pm)cryptopleurine (**174**) (58JA930; 64RTC593). The Westphal condensation of 1-ethoxycarbonylmethyl-2-methylpyridinium salt with phenanthrenequin-

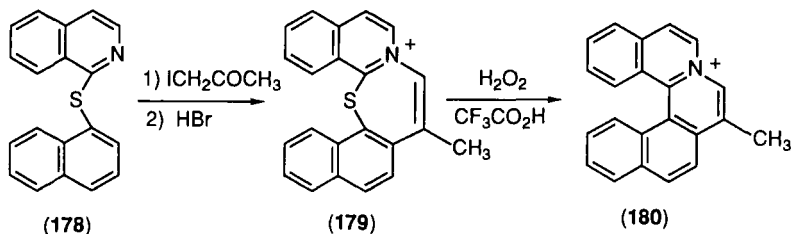
none in the presence of sodium bicarbonate or di-*n*-butylamine gave **30** (75%) (61AP37; 85JHC681; 86JHC1151).



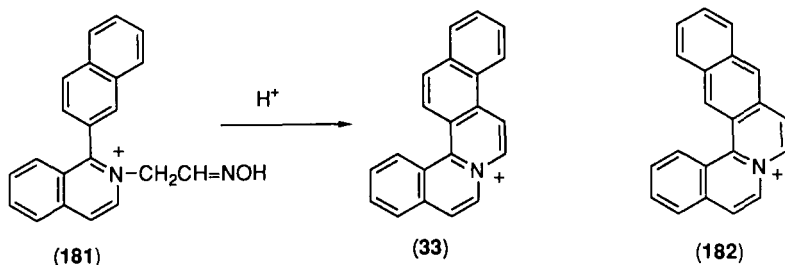
1-(2-Naphthyl)pyridinium salt **176** derived from 2-carboxy-4,6-diphenylpyrylium salt **(175)** was photocyclized in ethanol-chloroform to afford the 6-phenyl derivative **(177)** of **31** (63%) (84KGS1528).



e. *Azoniadibenzo[c,g]phenanthrene*. The oxidative sulfur extrusion of thiazepinium salt **179**, which was derived from the cyclodehydration of **178**, was carried out using hydrogen peroxide in trifluoroacetic acid to afford the azonia derivative of pentahelicene (**180**: 5-methylbenzo[*a*]naphtho[2,1-*h*]quinolizinium salt) (33% yield from **178**) [87JCS(P1)481].



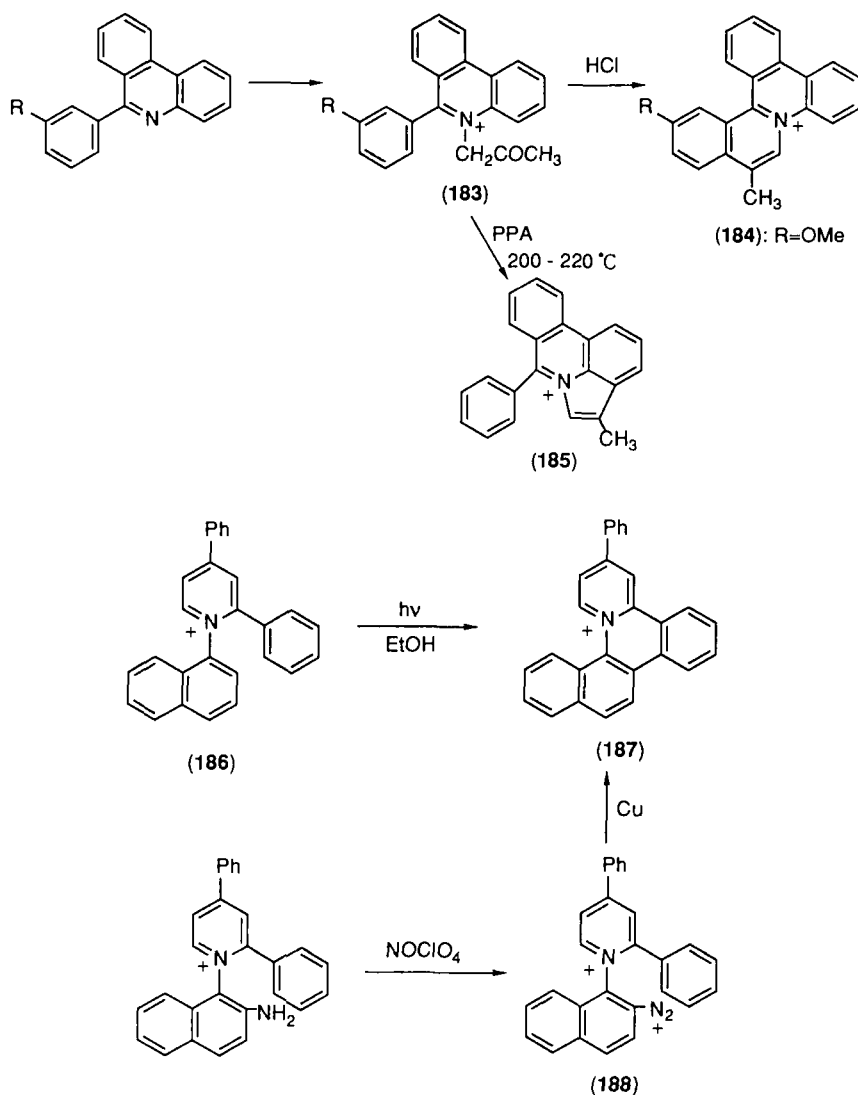
f. *Azoniabenzof[c]chrysene*. The cyclodehydration of salt **181**, derived from 1-(2-naphthyl)isoquinoline and bromoacetaldoxime, occurred at position 1 of the naphthalene ring to give benzo[*a*]naphtho[1,2-*h*]quinolizinium salt (**33**) (51%). The structure was proposed to be not **182**, but **33** on the basis of UV data (65JOC1846).



g. *Azoniadibenzo[*a,c*]phenanthrenes*. Four isomeric derivatives of this system have been reported. The cyclodehydration of salt **183** was unsuccessful, but by introduction of a methoxy group at a meta-position, the salt was cyclized with refluxing conc. HCl to afford 14-methoxy-11-methylisoquino[2,1-*f*]phenanthridinium salt (**184**) (55%) (59JOC592). When the cyclization was carried out under very vigorous conditions (200–220°C in PPA), the cyclization occurred at position 1 of the naphthalene ring to afford **185** (64JHC208).

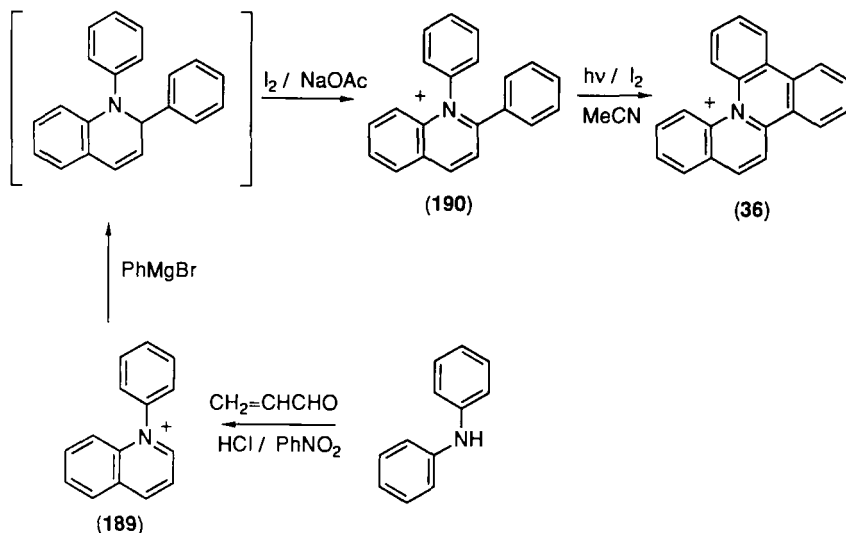
The synthesis of 3-phenyl derivative **187** of benzo[*c*]pyrido[1,2-*f*]phenanthridinium salt (**35**) has been achieved through two routes: photocyclization and a Pshorr reaction. The photocyclization of 1-naphthylpyridinium salt **186** in ethanol afforded **187** (76%) (84KGS1528). The intramolecular coupling reaction of diazonium salt **188** using Cu powder also gave **187** (74KGS1344).

1-Phenylquinolinium ion (**189**), obtained under Skraup conditions, reacted with a Grignard reagent and subsequently oxidized to yield 1,2-diphenylquinolinium salt (**190**). An acetonitrile solution of **190** was irradi-



ated in the presence of iodine to yield quino[1,2-*f*]phenanthridinium salt **(36)** (50%) (78T363). The isomeric compound **37** was also reported (74KGS1344; 84KGS1528).

h. Azoniabenzo[*e*]pyrenes. The synthesis of phenyl derivatives of quinolizino[3,4,5,6-*def*]phenanthridinium salt **(38)** was achieved through



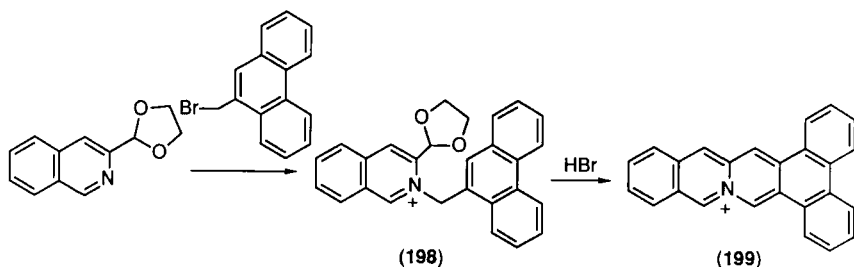
photocyclization by using three different types of starting compounds (89CS367). A methanolic solution of 1,4,6-triphenyl-2-styrylpyridinium salt (**191**) was irradiated in the presence of hydrogen iodide to afford **192** (30%). The photocyclization of **193** and 1,3-diphenylbenzo[*c*]quinolizinium salt (**194**) in the presence of triethylamine gave **192** (93.5%) and **195** (80%), respectively.

i. *Azoniaperylene*. 2-(1-Naphthyl)-6-methylpyridine **196** (10%) was obtained by the reaction of picoline *N*-oxide with the Grignard reagent prepared from 1-bromo-8-(methoxymethyl)naphthalene. Pyridine **196** was treated with HBr - AcOH to afford the cyclization product **197** (55%). The Westphal condensation of **197** with 2,3-dihydroxy-1,4-dioxane in the presence of triethylamine gave 12*b*-azoniaperylene salt (**39**) (70%) (91JOC-4858).

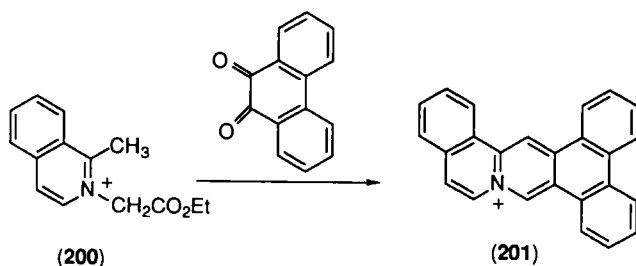
5. Polycyclic Aromatic Nitrogen Cations

a. *Benzo[b]phenanthro[9,10-*g*]quinolizinium Salt (199)*. Quaternization of 3-(1,3-dioxolan-2-yl)isoquinoline with 9-bromomethylphenanthrene yielded salt **198**, which was cyclized in boiling HBr to yield **199** (68JOC390).

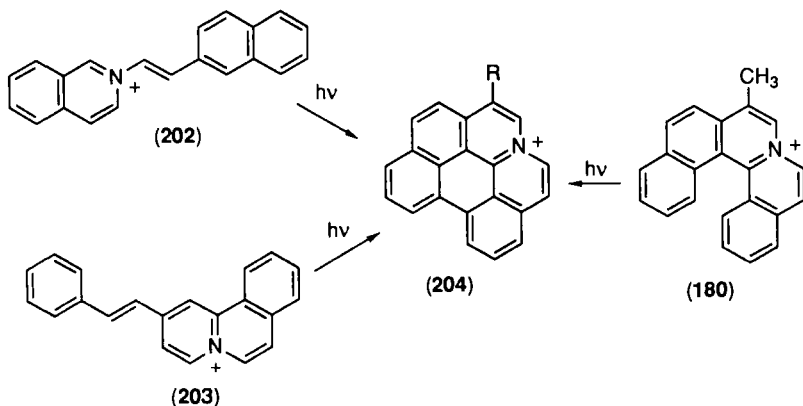
b. *Benzo[a]phenanthro[9,10-*g*]quinolizinium Salt (201)*. The Westphal condensation was applied to the synthesis of this system. The reaction



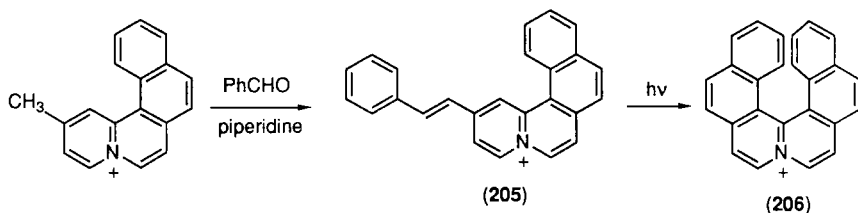
of 1-ethoxycarbonylmethylisoquinolinium salt (**200**) with phenanthrenequinone in the presence of dibutylamine or anhydrous sodium acetate yielded **201** (85%) (61AP37; 86JHC1151).



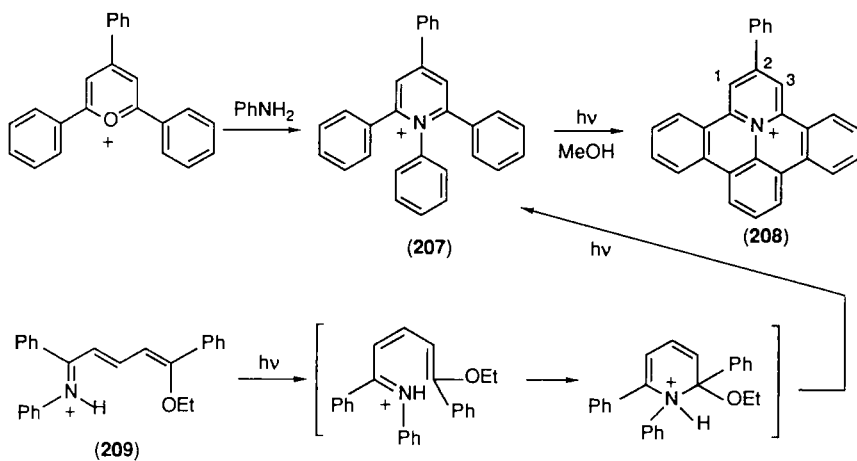
c. *Benz[4,10]anthra[1,9,8-hija]quinolizinium Salts (204)*. The photocyclization of 2-[2-(2-naphthyl)vinyl]isoquinolinium salt (**202**) and 2-styrylbenzo[*a*]quinolizinium salt (**203**) gave **204** ($R = H$) in 30% and 66% yields, respectively [87JCS(P1)481; 91BCJ1996]. Irradiation of compound **180** also gave methyl derivative **204** ($R = Me$). The 7-aza derivative was also obtained (91BCJ1996).



d. *Dinaphtho[1,2-a:2',1'-h]quinolizinium Salt (206)*. The first azonia derivative (**206**) of hexahelicene was synthesized by the photocyclization of 2-styrylnaphtho[1,2-a]quinolizinium salt (**205**) in 13% yield (89TL7217).

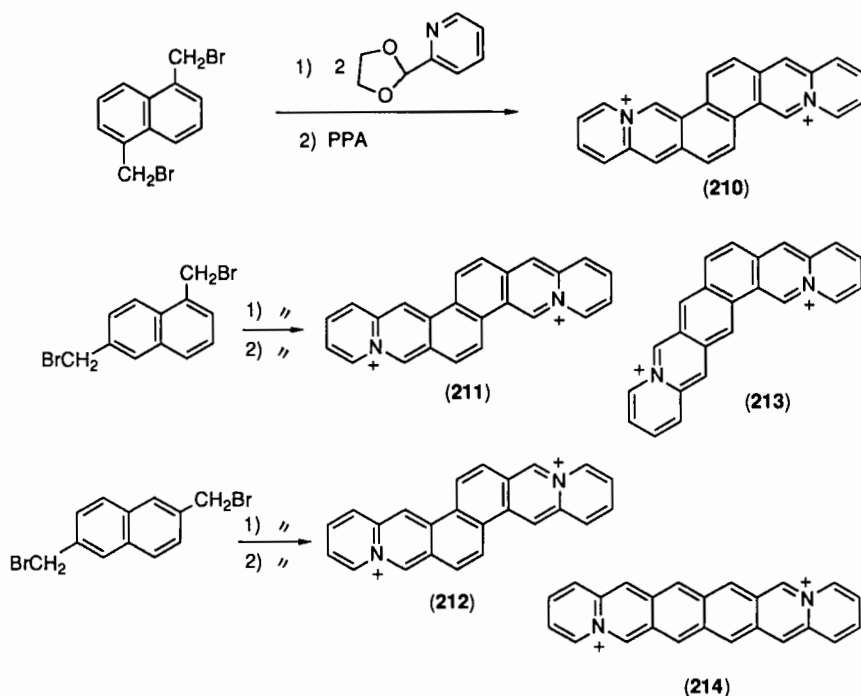


e. *Benzo[1,2]quinolizino[3,4,5,6-def]phenanthridinium Salt (208)*. The UV irradiation of a methanolic solution of 1,2,4,6-tetraphenylpyridinium salt (**207**) yielded **208** (85%) by double photocyclization. Many derivatives were also synthesized [80JCS(P1)1879]. Compound **208** was observed to form a pseudo-base on the basis of NMR measurements (83OMR649). Katritzky and co-workers also reported the preparation of a thieno fused compound (84KGS1509). An ethanolic solution of **209** was irradiated to afford **208** (40%) (89ZOR2603). Irradiation of 1-(4-pyridyl)-2,4,6-triphenylpyridinium salt caused two successive photocyclizations to afford an aza derivative (81ZOR610).



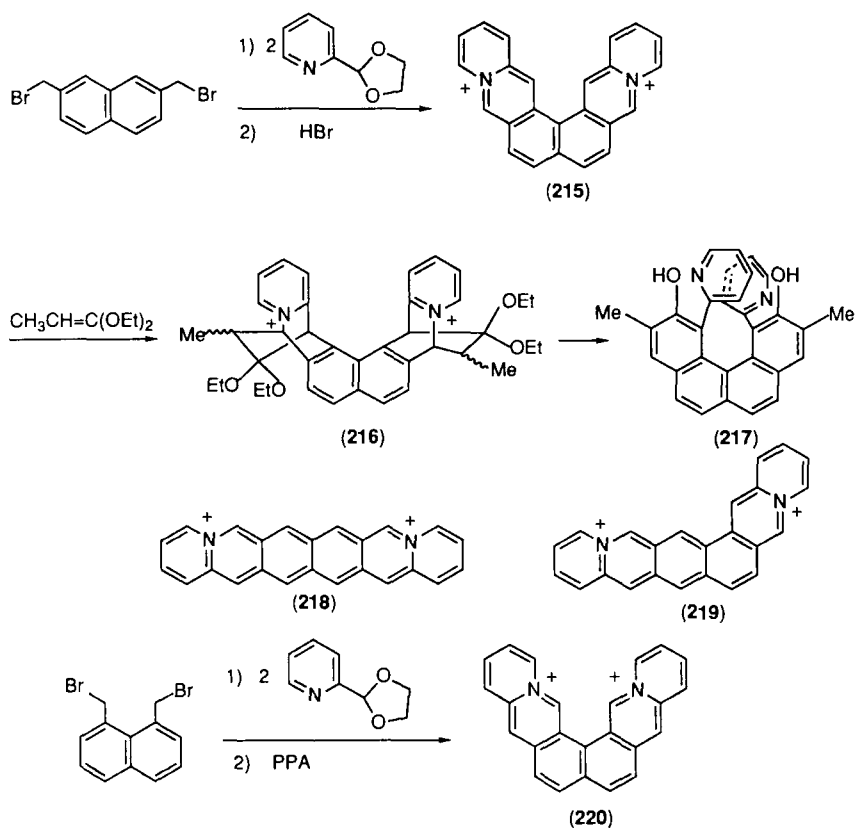
f. *Diazoniadibenzo[b,k]chrysenes*. Cyclodehydration was applied to the synthesis of these hexacycles. Starting from 1,5-, 1,6-, and 2,6-dibromomethylnaphthalenes, three diazonia derivatives of dibenzo-

[*b,k*]chrysene, naphtho[2,1-*b*:6,5-*b'*]diquinolizinediium salt (**210**), naphtho[1,2-*b*:6,5-*b'*]diquinolizinediium salt (**211**), and naphtho[1,2-*b*:5,6-*b'*]diquinolizinediium salt (**212**) were prepared in 85, 34, and 54% yields, respectively (68JHC253). On the basis of UV spectra, the isomeric structures **213** and **214** were rejected. These hexacycles are rather unstable. On addition of bicarbonate, an aqueous solution turned blue then brown. Bradsher described the fact that even polar solvents attacked the azonia compounds and the color turned from yellow to blue.

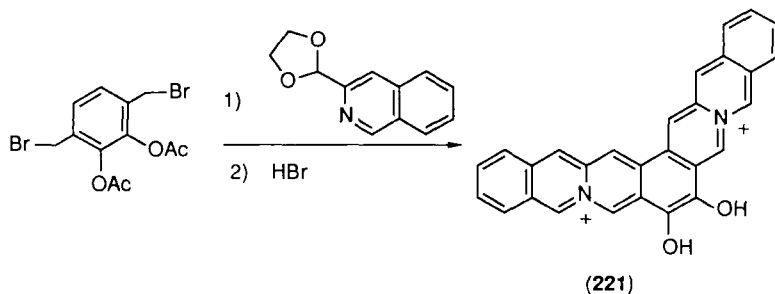


*g. Diazoniaanthra[1,2-*a*]anthracenes.* The reaction of 2,7-bis (bromomethyl)naphthalene and 2-(1,3-dioxolan-2-yl)pyridine gave the salt, which was cyclized on treatment with acid to yield naphtho[1,2-*b*:8,7-*b'*]diquinolizinediium salt (**215**; 90%) (73JHC195). The reaction of **215** with 1,1-diethoxypropene gave adduct **216**, which was converted to **217** by treatment with hydrochloric acid. This result supports the conclusion that the cyclization product is neither linear **218** nor angular **219**.

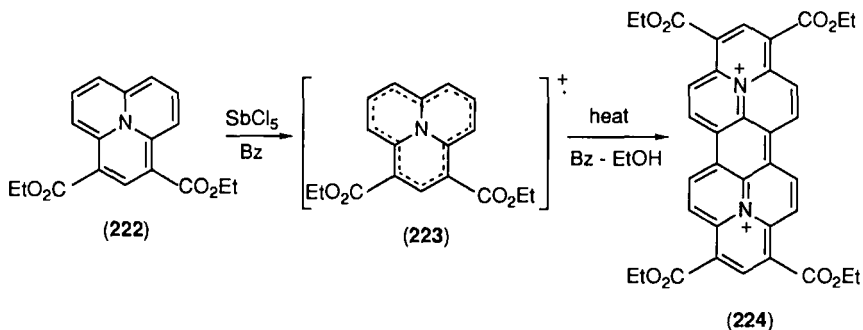
By using 1,8-bis(bromomethyl)naphthalene, naphtho[2,1-*b*:7,8-*b'*]diquinolizinediium salt (**220**) was obtained (90%) (68JHC253).



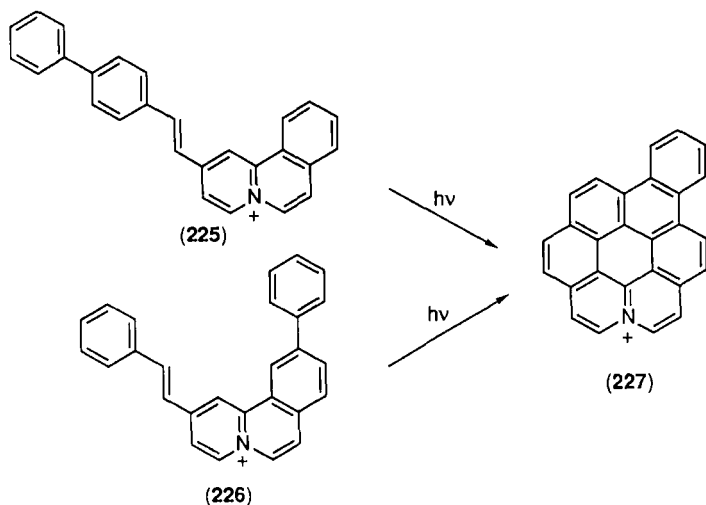
h. *Diisoquino[3,2-b:2', 3'-J][3,8]phenanthroline*dium Salt. Heptacyclic diazonia compound **221** was prepared in 56% yield by cyclodehydration of the quaternary salt from 3,6-bis (bromomethyl)catechol diacetate and 3-(1,3-dioxolan-2-yl)isoquinoline (65JOC252). This compound is insoluble in common organic solvents.



i. *Diquinolizino*[6,5,4,3-cde:3',4',5',6'-ghi][4,7]*phenanthridinediium Salt*. Cyclazine diester **222** was treated with antimony pentachloride in benzene to give a deep blue precipitate, presumed to be radical cation salt **223**. This blue solid was treated with hot benzene-ethanol to give bright red diazoniadibenzoperylene salt **224** [76JCS(P1)341]. Oxidative dimerization takes place easily because of the low ionization potential of cycl[3,3,3]-azine **222**.

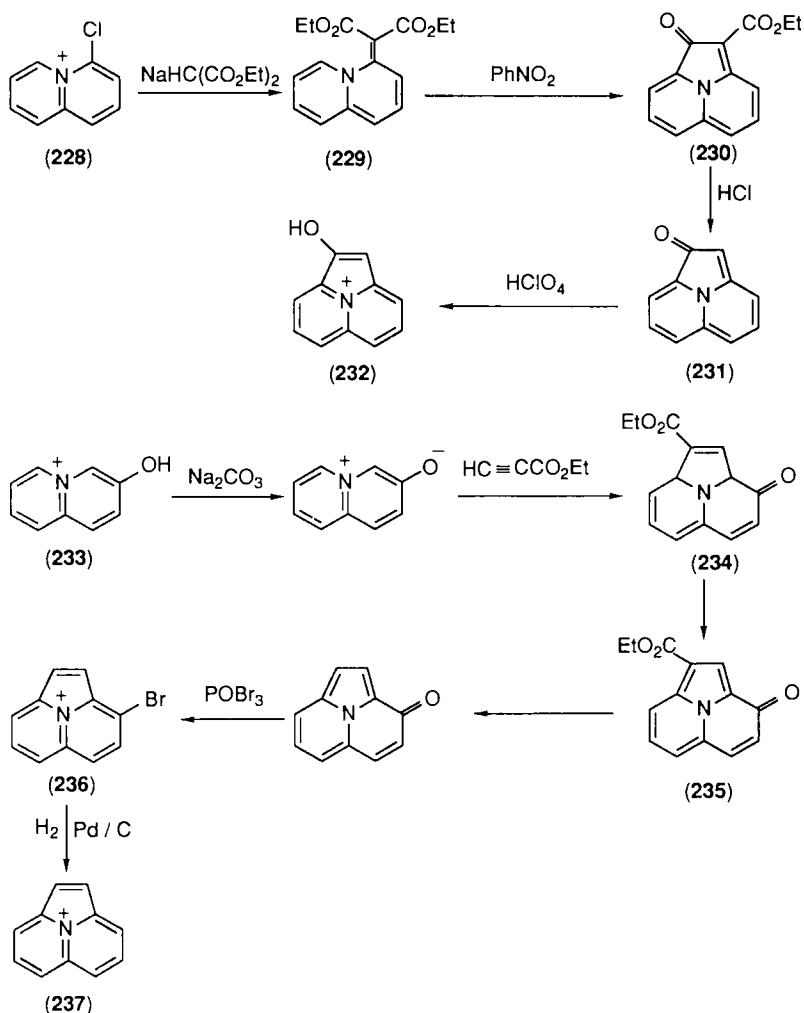


j. *2a-Azoniabenz[a]coronene*. Photocyclization of salts **225** and **226** in methanol yielded 2a-azoniabenz[a]coronene salt (**227**) via three successive photocyclizations in 80% and 75% yields, respectively (91BCJ1996).



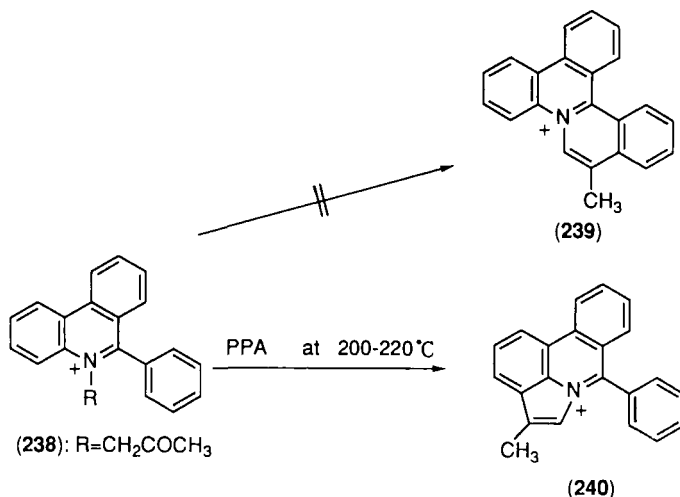
B. NONBENZENOID AROMATIC NITROGEN CATIONS

The azonia derivative of acenaphthylene was obtained by two routes [84JCS(P1)2553]. 4-Chloroquinolizinium salt (**228**) was reacted with diethyl sodiomalonate to afford diethyl quinolizin-4-ylidenemalonate (**229**). The quinolizine **229** was cyclized on treatment with refluxing nitrobenzene to afford cyclazinone **230** (75%), which was deethoxycarbonylated to give **231**. 1-Hydroxypyrrolo[2,1,5-*de*]quinolizinium salt (**232**) was prepared by treating the resulting cyclazinone **231** with perchloric acid.

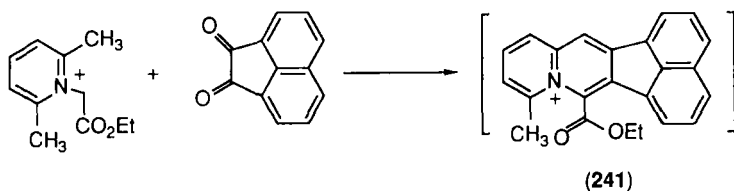


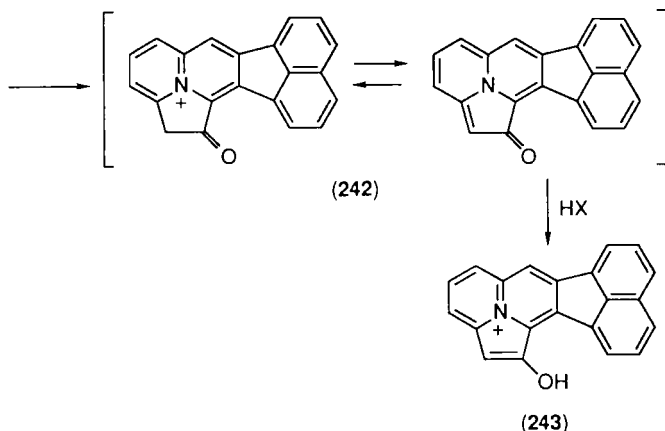
Another route used 3-hydroxyquinolizinium salt (**233**) as a starting compound. The reaction of **233** with ethyl propiolate in the presence of sodium carbonate in boiling nitrobenzene gave **234**, which was dehydrogenated to afford cyclazinone **235** (75%). The ketone **235** was decarboxylated and then treated with phosphoryl bromide to yield 3-bromocyclazinylium salt **236** (70%). Parent compound **237** was obtained by catalytic hydrogenation of **236** (74%).

5*a*-Azoniaacephenanthrylene was prepared by cyclodehydration. 5-Acetonyl-6-phenylphenanthridinium salt (**238**) was heated in phosphoric acid at 200–220°C to afford not 11-methyldibenzo [*a,c*]phenanthridinium salt (**239**), but 5*a*-azoniaacephenanthrylene salt (**240**) (29%) (64JHC208).

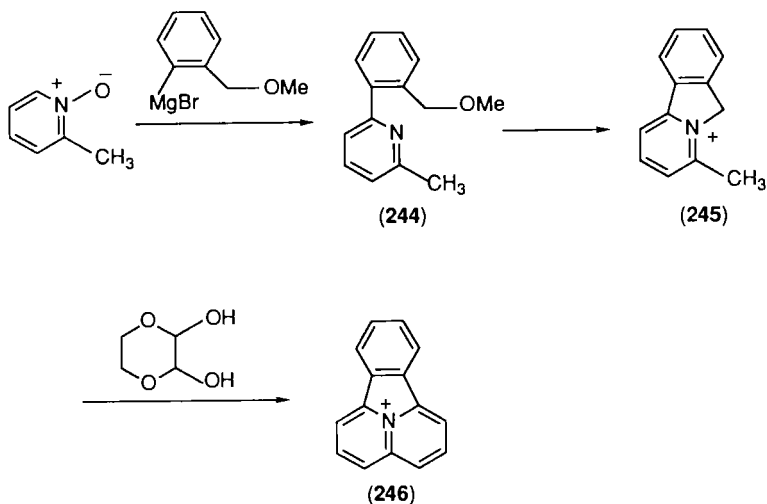


Fused acenaphthylene derivative was prepared (52%) by Westphal condensation (89H2369). The reaction of 1-ethoxycarbonylmethyl-2,6-dimethylpyridinium salt and acenaphthenequinone in the presence of dibutylamine gave quinolizinium salt **241**. Intramolecular Claisen reaction of **241** occurred under the reaction conditions to afford cyclazinone **242**, which was treated with HBr to yield **243**.



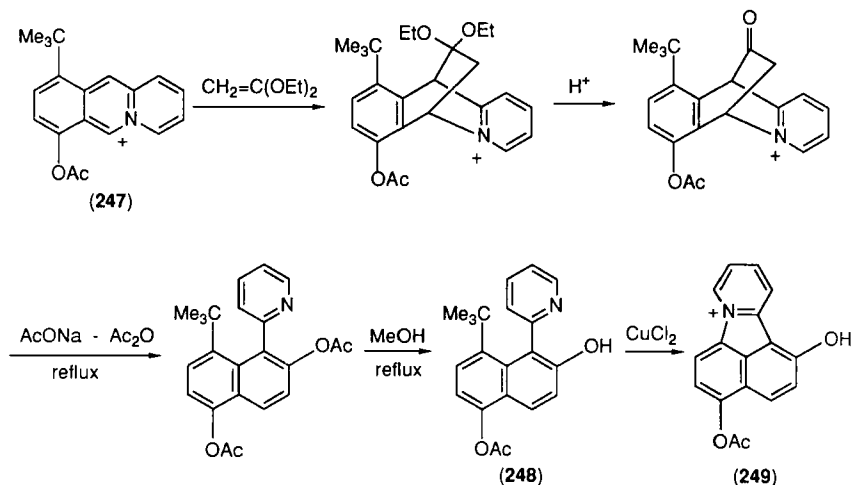


Two isomers of azoniafluoranthene have been reported. The reaction of 2-methylpyridine *N*-oxide with a Grignard reagent gave the pyridine **244** which, on treatment with HBr, cyclized to **245**. The Westphal condensation between **245** and 2,3-dihydroxy-1,4-dioxane in the presence of triethylamine yielded 10*c*-azoniafluoranthene salt (**246**) (33%) [89AG(E)588].

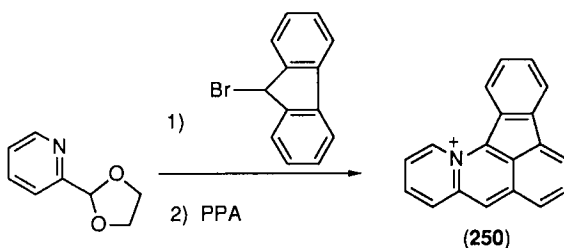


The cycloaddition of ketene diethyl acetal to benzo[*b*]quinolizinium salt **247**, followed by hydrolysis and thermolysis, gave over-crowded 1-pyridyl-8-*tert*-butylnaphthalene **248**. The naphthalene **248** was oxidized electrochemically or with anhydrous CuCl_2 to afford azoniafluoranthene salt **249**,

which was converted to a zwitterion by treatment with base (71JOC2986; 72JOC3058).

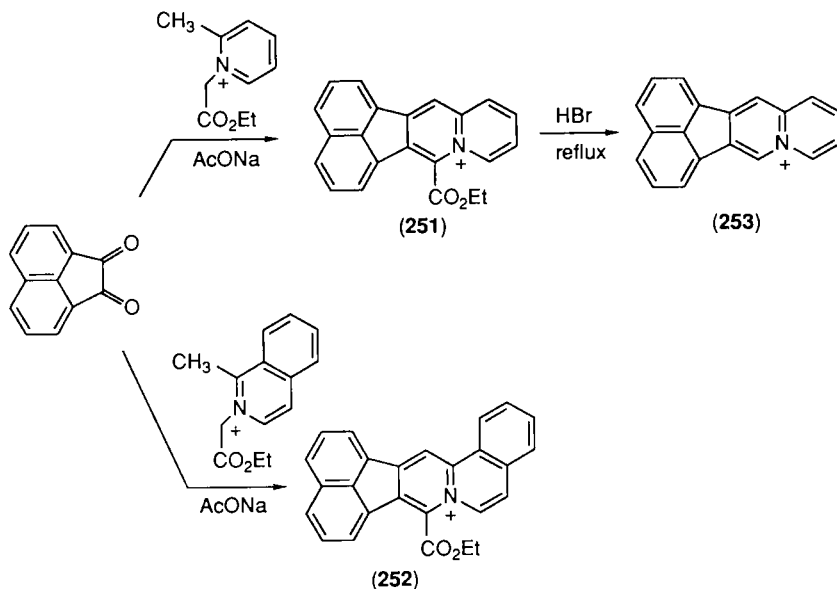


The reaction of 2-(1,3-dioxolan-2-yl)pyridine with 9-bromofluorene gave the quaternary salt (41%), which was cyclized in polyphosphoric acid at 110–120°C to afford 12a-azoniabenzofluoranthene salt (250) (64JHC121).

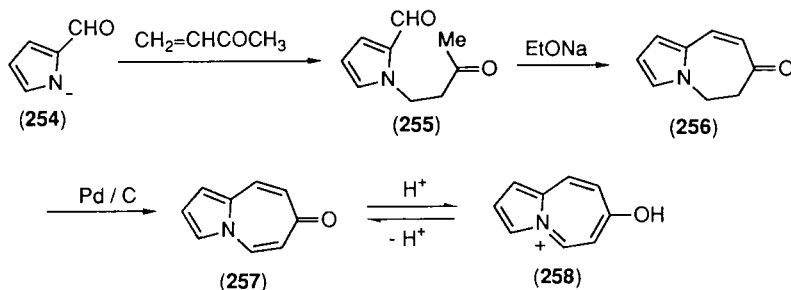


Westphal condensation of acenaphthenequinone with 1-ethoxycarbonylmethyl-2-methylpyridinium salt and 2-ethoxycarbonylmethyl-1-methylisoquinolinium salt in the presence of sodium acetate gave **251** (73%) and **252** (44%), respectively (86JHC1151). On heating **251** with HBr , parent compound **253** was obtained (48%) (85JHC681; 86JHC1151; 89H2369).

Protonation of pyrrolo[1,2-*a*]azepin-7-one **257** gave hydroxyazoniaazulene. The yellow solution of **257** turns deep blue. ^1H -NMR spectroscopy shows a downfield shift of ring protons, and the coupling constants are reduced and closer to those for azulene [82JCS(P)1123]. Jones and Radley reported the synthesis of the pyrroloazepinone **257**. The reaction of pyr-

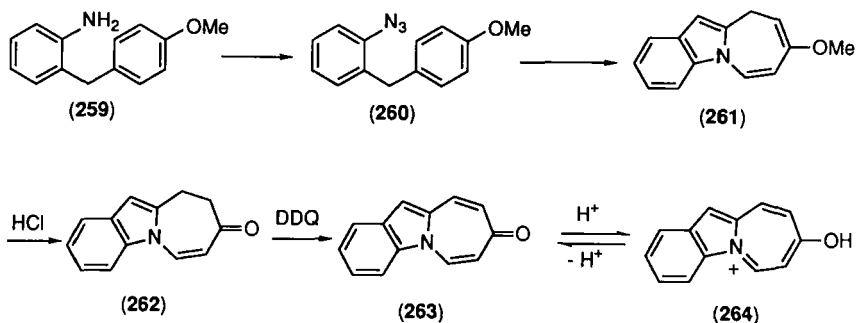


role-2-carbaldehyde anion (**254**) with but-3-en-2-one gave ketone **255** (68%), which underwent an intramolecular aldol reaction to afford dihydropyrroloazepinone **256** (40%). The dehydrogenation of **256** with palladium on charcoal yielded the pyrroloazepinone **257** [82JCS(P1)1123]. Although parent azoniaazulene has not been reported, substituted pyrroloazepinones were prepared [69JCS(C)1028; 78CB2407; 87S262].

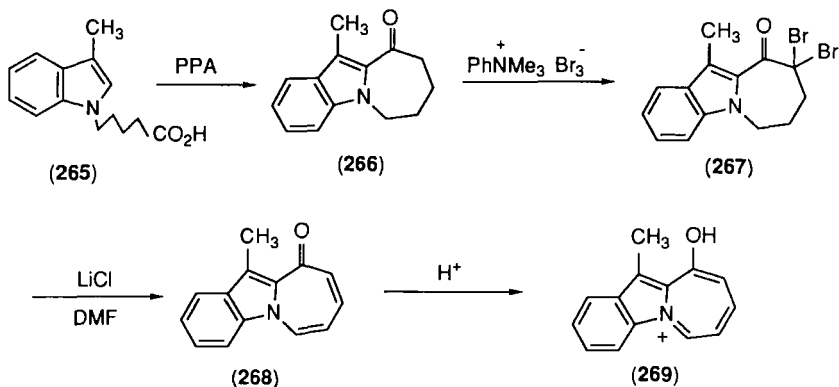


Benzo-fused azoniaazulenes were also reported. *o*-Benzylphenyl azide **260** (92%) was obtained by diazotization of **259** followed by treatment with sodium azide. Decomposition of the azide **260** at 190°C gave azepinoindole **261**, which was hydrolyzed to afford dihydroazepinoindolone **262** (90%). Dehydrogenation of **262** with dichlorodicyanobenzoquinone yielded azepi-

noindolone **263** (80%), which was treated with HBr to give hydroxyazepinoindolium salt **264** [71JCS(C)3418].



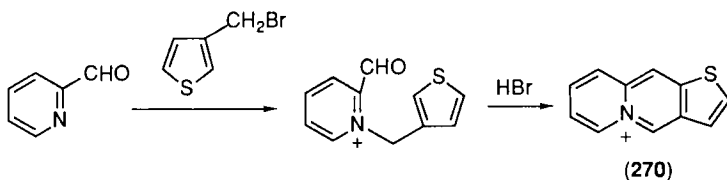
Jones also reported the synthesis of hydroxyazepinoindolium **269** (68TL1935). *N*-Substituted indole **265** was cyclized to yield ketone **266**. Ketone **266** was reacted with perbromide to give dibromoketone **267**, which was treated with lithium chloride in DMF to yield **268**. Ketone **268** was converted to **269** by addition of acid.



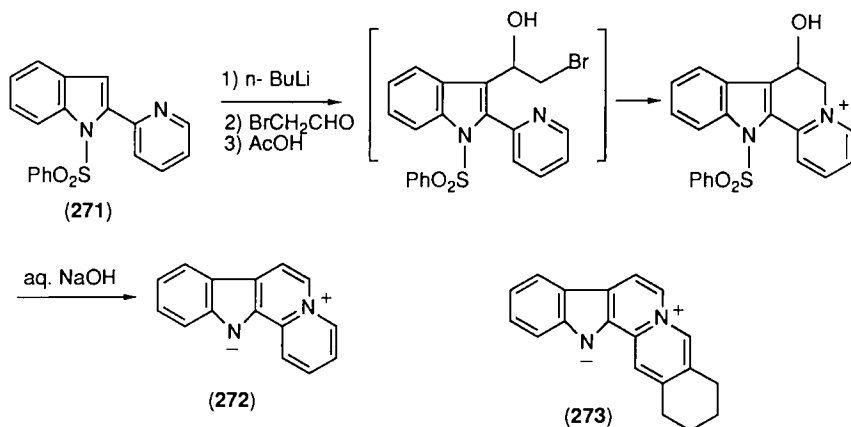
C. AROMATIC NITROGEN CATIONS FUSED WITH A π -SUFFICIENT HETEROCYCLE

In this section, the synthesis of quinolizinium ions fused with π -sufficient heteroaromatics, such as thiophene and indole, is described.

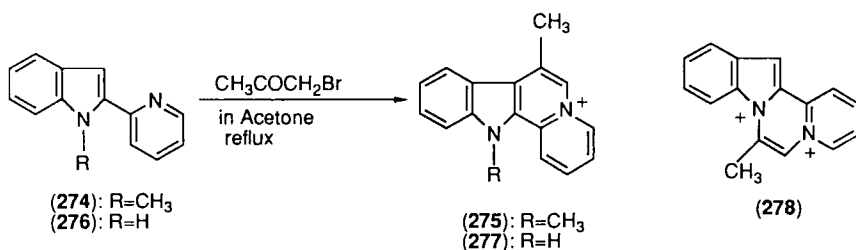
Quinolizinium ions fused with thiophene were prepared by cyclo dehydration. A quaternary salt formed by reaction between 2-pyridinecarbaldehyde and 3-bromomethylthiophene was cyclized with HBr to thieno[3,2-*b*]quinolizinium salt (**270**) (72%) (57JA4380).



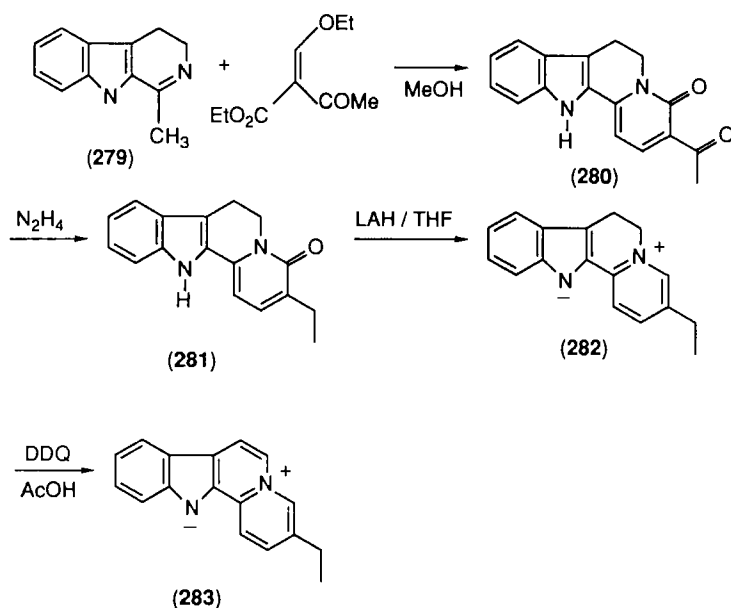
Quinolizinium ions fused with indole are related to yohimbine alkaloids: flavopereirine, sempervirine, reserpine, alstonine, ajmaline, and so on. Parent indolo[2,3-*a*]quinolizinium salt (**272**) has been reported (87TL5259). 1-Phenylsulfonyl-2-(2-pyridyl)indole **271** was treated with *n*-butyllithium to afford the 3-lithio species, which was quenched with bromoacetaldehyde to give the cyclization product (48%). It was dehydrated with aq. NaOH to afford **272** (89%). Using this method, sempervirine (**273**) was obtained (88T3195).



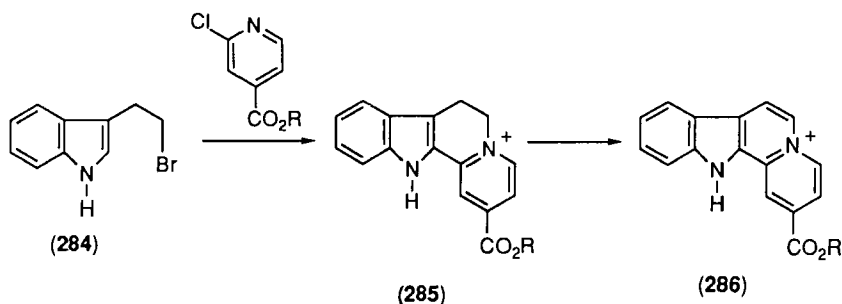
The reaction of 1-methyl-2-(2'-pyridyl)indole (**274**) with bromoacetone in acetone gave the dimethyl derivative (**275**) of indoloquinolizinium salt (42%). The cyclization product from 2-(2'-pyridyl)indole (**276**), however, was not 7-methyl-12*H*-indolo[2,3-*a*]quinolizinium salt (**277**), but 11-methylindolo[2,1-*a*]-2-azoniaquinolizinium salt (**278**) (34%) (64JOC3584).



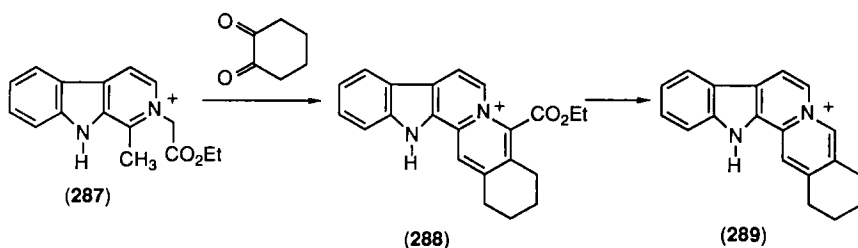
Flavopereirine (**283**), which is the ethyl derivative of **273**, was synthesized by the reaction of **279**–**283**. The condensation of 1-methyl-3,4-dihydro- β -carboline (**279**) and ethyl ethoxymethyleneacetoacetate gave diketone **280**, which was reduced under Huang–Minlon conditions to afford **281**. Amide **281** was reduced with LAH then oxidized with DDQ to afford **283** (84H233).



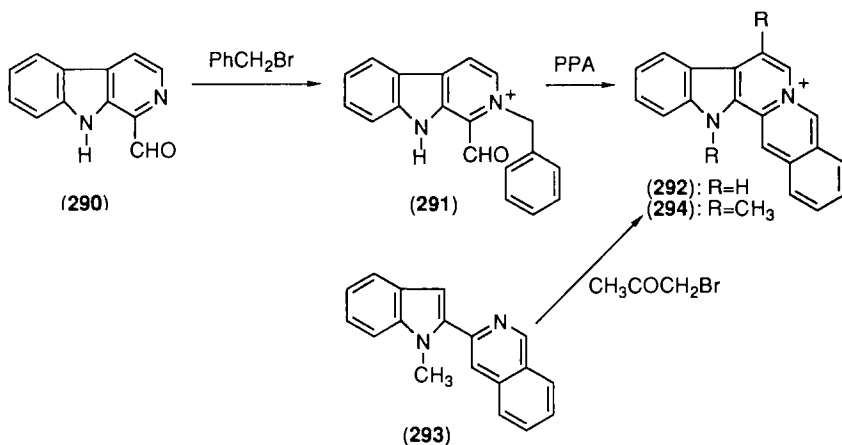
The reaction of 3-(2-bromoethyl)indole (**284**) with 2-chloropyridine gave indoloquinolizine **285**, which was dehydrogenated with tetrachloro-*o*-benzoquinone to yield indoloquinolizinium salt **286** (62JA3393; 68CPB549).



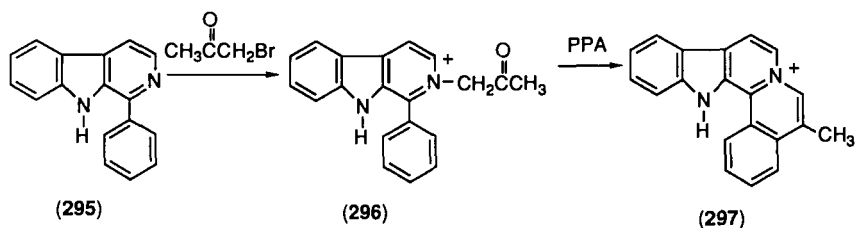
The Westphal condensation was applied to the synthesis of sempervirine (**289**) (68JOC3985). Quaternary salt **287** reacted with 1,2-cyclohexanedione in the presence of dibutylamine to give quinolizinium salt **288** (48%), which was hydrolyzed (93%) and decarboxylated (85%) to afford **289**.



Fully aromatic sempervirine **292** was also synthesized. The reaction of pyridoindolecarbaldehyde **290** with benzyl bromide gave quaternary salt **291**, which was cyclized under acidic conditions to give indolo[2,3-*a*]acridizinium salt (**292**) (72%) with a strong yellow-green fluorescence (63JOC3070). Refluxing isoquinolyindole **293** with bromoacetone in acetone yielded **294** (58%) (64JOC3584).



Benzo[*a*]quinolizinium **297** with a fused indole ring was synthesized (17%) by the cyclodehydration of the quaternary salt **296**, obtained (95%) from 1-phenylpyridoindole **295** and bromoacetone (64JHC168).



V. Structures and Physical Properties

A. STRUCTURES

Single crystal X-ray studies of the parent bicyclic quinolizinium and tricyclic benzoquinolizinium salts have not been reported. Two examples are available: 9-phenylbenzo[*b*]quinolizinium salt (**74**) (90MI1) and 10*c*-azoniafluoranthene salt (**246**) [89AG(E)588]. In the case of planar cation **246**, a significant contraction of the distances was observed around the quaternary nitrogen atom compared to fluoranthene.

Wang and Jones studied solid-state chemistry of compound **2** and found that irradiation of single crystals of **2** gave the single crystals of the photodimer (87T1273).

B. SPECTROSCOPIC PROPERTIES

1. Electronic Spectra

a. *Theoretical Treatment of Electronic Spectra.* The light absorption of aromatic compounds arises from a transition of the electronic state of the π -electron system. The position and strength of the light absorption correspond to the transition energy and the transition probability (oscillator strength), respectively. The transition energy and the oscillator strength can be calculated using MO theory in the following manner.

i. *Step 1.* The molecular orbitals can be calculated by a variety of MO methods. Although HMO is a simple and convenient method, it is not suitable for calculation of the light absorption because of the neglect of electron repulsion, except for cases of cyanine and cationic dyes (50JCS2329). The PPP method has been widely used among advanced MO theories in the calculation of the light absorption of π -electron systems. To use the PPP method, the determination of the coordinates of atoms

and some energy parameters is required. The coordinates of atoms are determined using crystallographic data, if possible, or by assuming a regular polygonal structure with a bond length of 1.39 Å for C—C bonds.

Selection of correct energy parameters, ionization potentials, and electron repulsion integrals is very important because these values largely influence the calculated results. The values for carbon and nitrogen atoms currently used are determined by the ionization potential and electron affinity in each valence state (62JA540). The parameters for the nitrogen cation vary somewhat. Brown suggested that the coulomb integral of a nitrogen cation is 3β , based on a comparison between PPP and CNDO calculations of a $C=N^+$ system (57TFS397). Ohno proposed the parameters on a theoretical basis: $I_p(N^+) = 29.55$ eV and the electron repulsion ($N^+N^+ | N^+N^+$) = 23 eV (64TCA219). Calculated results for quinolizinium cation (**1**), using the different values of the parameters, are compared in Table VII.

ii. *Step 2.* The electronic configuration with all the electrons in the lowest energy molecular orbitals has the lowest energy. Electron configurations of higher energy are formed by promoting an electron from an occupied MO to an unoccupied MO. Among them, the electron configurations formed by the following transitions are responsible for the electronic spectra in the near UV and visible regions: A transition from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), a transition from the HOMO to the next LUMO, a transi-

TABLE VII
CALCULATED ABSORPTION SPECTRA OF **1** USING DIFFERENT VALUES OF PARAMETERS

	I ^a	II ^b	III ^c	Obs ^d
I_p^e (eV)	11.95	28.71	28.6	
($N^+N^+ N^+N^+$) ^f	11.07	VESCF ^g	12.34	
Position of	3.944	3.934	3.771	3.84
absorption	3.986	4.524	4.521	4.38
band (eV):	5.307	5.834	5.634	5.49
	5.921		5.661	

^a Ref. 68TCA(11)417.

^b Ref. 71TCA216.

^c M. Hida, unpublished results.

^d Ref. 54JA1832.

^e Ionization potential.

^f One-center coulomb repulsion integral at bridgehead nitrogen.

^g Variable electronegativity SCF method (57TFS397).

tion from the next HOMO to the LUMO, and further the transition from the next HOMO to the next LUMO.

iii. *Step 3.* The ground state of molecules can be adequately approximated by the lowest electron configuration. However, the wave functions of the excited states must be expressed by a linear combination of electron configurations of higher energy. This procedure is called configuration interaction (CI).

b. *Electronic Spectra of Alternant Aromatic Nitrogen Cations.* Alternant hydrocarbons have three rather well-defined absorption bands in the near UV and visible regions. These bands are related to the three bands of benzene; the first (the longest wavelength band) is called the α band, the second is the p band, and the third is the β band by Clar (52MI1). Interpretation of spectra has proceeded as follows.

These bands are related to the four transitions, HOMO–LUMO, next-HOMO–LUMO, HOMO–next-LUMO, and next-HOMO–next-LUMO. The electron configurations of next-HOMO–LUMO and HOMO–next-LUMO are degenerate, and they split into two components by the CI procedure, one of which lies below the transition of HOMO–LUMO with the lowest energy electron configuration. Therefore the lowest excited state (α band) is one component of the doublet pair (next-HOMO–LUMO and HOMO–next-LUMO). The second band (p band) corresponds to the transition of HOMO–LUMO. The third shortest wavelength and the strongest band (β band) is associated with the next HOMO–next-LUMO transition and another component of the doublet pair.

First-order perturbation theory within the simple MO method derives the following generalizations for changes in spectra by substituting a nitrogen for a carbon atom in an alternant hydrocarbon.

- (1) Both energies of the HOMO–LUMO and next-HOMO–next-LUMO transitions are roughly unchanged, and the doublet pair (the next-HOMO–LUMO and HOMO–next-LUMO transitions) splits into two levels.
- (2) By the CI procedure, HOMO–LUMO and next-HOMO–next-LUMO transitions become very similar to the p and β bands of the parent hydrocarbon, respectively, while the pair of next-HOMO–LUMO and HOMO–next-LUMO will become α and β' bands with a little longer wavelength than those of the parent hydrocarbon. In Table VIII, the calculated maximum wavelengths are compared with the observed values. The weights of the component configurations by the CI procedure are also summarized in the table.

TABLE VIII
ABSORPTION SPECTRA OF ALTERNATE HYDROCARBONS AND AROMATIC NITROGEN
COMPOUNDS AND WEIGHTS OF COMPONENT CONFIGURATIONS BY THE CI PROCEDURE^a

Compound	Transition energy (nm)	Excitation energy (nm) after CI	Configuration: weight (%)	Obs. (nm)
Benzene	208(2-5;3-4)	252	2-5:50, 3-4:50	250
	187(2-4;3-5)	200	2-4:50, 3-5:50	203
		170	2-4:1.8, 2-5:48.2 3-4:48.2, 3-5:1.8	
		170	2-4:48.2, 2-5:1.8 3-4:1.8, 3-5:48.2	180
Pyridine	232(3-4)	264	3-4:71, 2-5:29	259
	198(2-5)	206	3-5:35, 2-4:65	203
	195(2-4)	180	3-4:29, 2-5:71	176
	186(3-5)	177	3-5:65, 2-4:35	
Naphthalene	255(5-6)	297	5-7:48.7, 4-6:48.7	311
	245(4-6)	266	5-6:90, 4-7:9	275
	(5-7)			
	202(3-6)	215	<i>b</i>	220
1	(5-8)			
	199(4-7)	214	5-7:49.2, 4-6:49.2	
	309(5-6)	328	5-6:90, 5-8:1.1 4-7:7.6	323
	254(5-7)	274	5-7:70.9, 4-6:27.8	283
	227(4-6)	220	5-7:28.3, 4-6:62.9	226
	212(5-8)	219	5-6:6.3, 5-8:63.9 4-7:28	

^a M. Hida, unpublished data.

^b None of the transitions, HOMO–LUMO, next-HOMO–LUMO, HOMO–next-LUMO, and next-HOMO–next-LUMO, contributes to this absorption band energy.

- (3) The outstanding feature of replacing a carbon by a nitrogen atom is an increase in the strength of an α band. As stated previously, in the case of the alternant hydrocarbons, the degenerate pair of transitions (HOMO–next-LUMO and next-HOMO–LUMO) splits into two components by the CI procedure. If u_1 and u_2 denote the transition moments of these two transitions, respectively, the transition moments of the two components (M_H and M_L) can be written as weighted sums of u_1 and u_2 as follows:

The transition moment of the higher energy component corresponding to the β' band is

$$M_H = au_1 + bu_2$$

The lower energy component corresponding to the α band is

$$M_L = bu_1 - au_2$$

where both a and b are positive constants.

In the case of the alternant aromatic hydrocarbons, $u_1 = u_2$ and $a = b$; hence, the transition moment of the α band is zero. Since the oscillator strength is proportional to the square of the transition moment, the α band is forbidden, and its absorption intensity is very weak. For alternant aromatic nitrogen compounds, however, the values of u_1 and a are not equal to those of u_2 and b , respectively; hence, its transition moment does not vanish. Hence, the α band is allowed, and its absorption strength is stronger than that of the parent hydrocarbon. But it is still rather weak, because the value of a is near to that of b , and the transition moment (M_L) is not so large.

Another feature of the α band of an aromatic nitrogen compound is the loss of vibrational structure because of the reduced symmetry and more complicated vibration of the molecule skeleton.

The absorption spectra of polycyclic aromatic nitrogen cations can be interpreted as mentioned previously. Table IX shows that the calculated wavelengths of maximum absorption by the PPP method are in fairly good agreement with the observed values. A large discrepancy between the calculated and the observed values, however, is observed in the case of **8**. Since the first bands of its isomers (**7**, **9**, **10**, **11**, and **12**) are observed at a maximum of ~ 400 nm, as expected by the PPP calculation, the reported wavelength of **8** seems to be rather unreliable.

The absorption spectra of benzo[*b*]quinolizinium and dibenzo[*a,g*]quinolizinium derivatives are listed in tables X and XI, respectively. Substituent effects, however, cannot be deduced because of the lack of systematic data and the unreliable reported data of unsubstituted **8**.

Absorption spectra of more than five-ring systems are compared in Table XII, where only the first and second (and third) bands are listed for simplicity. Pericondensation (**195**) to **2** shifts the absorption band more bathochromically than catacondensation (**30**) does. Cyclization of azonia-pentahelicene salt (**180**) to the corresponding azoniabenzo[*ghi*]perylene salt (**204**) shifts the maximum absorption wavelength about 18 nm longer, similar to the annelation to form azoniahexahelicene salt (**206**).

c. Electronic Spectra of Nonalternant Aromatic Nitrogen Cations. Contrary to the case of the alternant π -electron systems, general interpretations of the electronic spectra of nonalternant π -electron systems are impossible. But the individual assignment of the spectra of the hetero-

TABLE IX
ABSORPTION SPECTRA OF TRI- AND TETRACYCLIC AROMATIC NITROGEN CATIONS^a

Compound	Calculated		Observed λ_{\max} (nm) (log ϵ)	Refs. ^c
	λ_{\max} (nm)	f^b		
2	402	0.394	399 (3.93), 379.5 (4.01)	55JA4812
	342	0.220	361 (3.99)	
	288	0.382		
	258	0.400	270 (4.05)sh	
	245	0.139		
	238	0.872	242 (4.68)	
	222	0.197		
3	362	0.259	354 (4.10), 338 (3.96)	87JCS(P1)481
	340	0.262	323 (3.67)	
	277	0.716	278 (4.28), 269 (4.23)	
	256	0.120	254sh	
	238	0.041	238 (4.32)	
	228	0.735	223 (4.38)	
	222	0.173		
4	377	0.455	365 (4.16), 349 (4.03)	66JOC2346
	325	0.035	332sh (3.68)	
	277	0.185	295 sh (3.62), 280(3.98)	
	258	0.247	255 (4.42)	
	246	0.852	229 (4.27)	
	220	0.293		
	218	0.196		
5	530	0.275		
	380	0.272		
	340	0.357		
	301	0.992		
	282	0.189		
	275	0.697		
	268	0.009		
6	427	0.246	423 (4.37)	60JOC191 ^d
	423	0.628	400 (3.95)	
	319	0.525	338 (4.23)	
	307	0.118	285 (4.60)	
	304	0.059		
	255	1.820	264 (4.53)	
	253	0.111		
7	468	0.099	420sh	65JHC399 ^e
	372	0.358	388 (3.77), 372 (3.77)	
	317	0.873	308 (4.84)	
	295	0.408	297sh	
	278	0.087		
	267	0.221	260 (4.36)	
	248	0.132		
	247	0.142	240 (4.42)	

(continued)

TABLE IX (Continued)

Compound	Calculated		Observed	Refs. ^c
	λ_{\max} (nm)	f^b	λ_{\max} (nm) (log ϵ)	
8	407	0.595	507, 475, 403, 383	58JOC430
	363	0.020	364, 346	
	317	0.300	309	
	292	0.185	296	
	286	0.522	270	
	253	0.727	260	
	246	0.307		
9	405	0.251	405.5 (3.89), 384 (3.91)	56JA2459
	363	0.002	364 (3.91)	
	331	1.242	312.5 (4.55)	
	293	0.204	276 (4.57)	
	278	0.136		
	265	0.199		
	245	0.018		
	242	0.458		
	232	0.118		
	225	0.380	227.5 (4.52)	
	225	0.191	400 (3.75)	
10	475	0.191	400 (3.75)	66JOC3683
	373	0.263	385 (3.75), 365sh (3.69)	
	317	0.656	313 (4.58)	
	297	0.340	299 (4.38), 285sh (4.31)	
	275	0.015		
	270	0.237		
	249	0.021		
	245	0.603	246 (4.57)	
	245	0.603	246 (4.57)	
11	421	0.469	428, 405	66JOC3683
	347	0.115	358	
	318	0.024		
	301	0.602	290	
	279	0.756		
	258	0.167		
	245	0.281	240sh	
	245	0.281	240sh	
12	412	0.630	395 (4.34), 376 (4.22)	56JA2459
	362	0.021	358 (3.93)	
	312	0.441	317 (4.39)	
	295	0.350	305 (4.33)	
	287	0.179		
	258	0.538	274 (4.58)	
	248	0.027		
	237	0.084		
	232	0.268	233 (4.45)	
	232	0.268	233 (4.45)	
14	394	0.553	387 (4.29), 368 (4.16)	66JOC3683
	354	0.292	350 (3.92)	

TABLE IX (Continued)

Compound	Calculated		Observed	Refs. ^c
	λ_{\max} (nm)	f^b	λ_{\max} (nm) (log ϵ)	
16	329	0.008		
	271	0.331	278 (4.32)	
	266	0.662		
	245	0.223		
	238	0.585	241 (4.20)	
	233	0.150		
	227	0.316	220 (4.45)	
	397	0.437	387 (4.04)	87JCS(P1)481
	365	0.085	367 (3.98)	
	302	0.311	348 (3.94)	
	297	0.444	310 (4.37)	
	290	0.044		
	253	0.328	259 (4.43)	
	249	0.030		
	239	0.161	236 sh	
	231	0.103		
19	223	0.759	225 (4.38)	
	404	0.627	405 (4.26), 384 (4.11)	91CL1355
	355	0.013	366 sh	
	318	0.005		
	288	0.066	277 sh	
	263	0.444	268 (4.49)	
	253	0.687		
	246	0.564		
	229	0.014		

^a Hida's parameters (Table VII) were used for calculation.^b Oscillator strength.^c References for observed data.^d Data of 3-methoxy derivative.^e Data of 13-methyl derivative.

aromatic compounds to those of the parent hydrocarbons can be made using the perturbation method similar to the case of the alternant aromatic compounds. Three examples are shown here. Variation of the energy of the transition $i - j$ by the introduction of a nitrogen at position r can be described by Eq. (10) within the first order approximation. If $|C_{rj}|$ is larger than $|C_{ri}|$, the transition energy will decrease by substitution of nitrogen for carbon atom and vice versa.

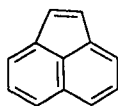
$$\Delta E_{i-j} = (C_{rj}^2 - C_{ri}^2)(\alpha_N - \alpha) \quad (10)$$

TABLE X
SUBSTITUENT EFFECT ON ABSORPTION SPECTRA OF BENZO[*b*]QUINOLIZINIUM SALTS

Substituents	λ max (nm)	$\Delta \sigma$ (10^3cm^{-1})	Refs.
H	388	0.00	55JA4812
4-Me	400	-0.06	63JOC83
6-Me	404	-0.31	64JHC121
7-Me	399.5	-0.03	55JA4812
9-Me	398	0.06	55JA4812
11-Me	402	-0.18	59JA2550
6-Ph	406	-0.43	67JOC733
11-Ph	404	-0.31	67JOC733
7-OMe		-1.56 ^a	
8-OMe	410	-0.67	57JA6033
9-OMe		-0.86 ^a	
6-NH ₂	405	-0.37	67JOC733
6-CN	432	-1.91	59JA2550
7,8-diOMe	438	-2.23	57JA6033
8,9-diOMe	396	0.19	57JA6033
6-Me-11-Ph	411	-0.62	59JA2550
7-Me-11-Ph	412	-0.79	59JA2550
9-Me-11-Ph	403	-0.25	59JA2550
8-OMe-11-Ph	420	-0.98	59JA2550

^a Calculated shift by assuming additivity of the substituent effect.

i. *Example 1: Acenaphthylene (298) and its azonia derivative (237).* The spectrum of **237** is very similar to that of **298**, except for the disappearance of the longest wavelength absorption band of **298**. The transition energies and the absorption band energies of **298** and **237** are compared in Table XIII. The shifts of the transitions estimated by Eq. (10) are also shown in the table. As shown by the arrow in the table, the calculated results suggest that the first and the second bands (x bands) of **298** will shift largely hypsochromically, but other bands will shift rather bathochromically. The disappearance of the first band of **298** is understood to be the result of its hypsochromic shift to the region of the second band of **237**.



(298)

TABLE XI
ABSORPTION SPECTRA OF DIBENZO[*a,g*]QUINOLIZINIUM SALTS

No.	Position	Substituents	$\lambda_{\max}(\text{nm})(\log \epsilon)^a$	Refs.
0		H	507, 475, 403	58JOC430
1	13	Ph	406 (4.25), 387 (4.15)	59JOC589
2	10	OMe	417, 397	58JOC430
3	9, 10	Me, Ph	416 (3.84), 396 (3.76)	59JOC589
4	10, 13	OMe, Ph	421 (4.03), 400 (3.93)	59JOC589
5	9, 10	(OMe) ₂	504, 473	58JOC430
6	9, 10	—OCH ₂ O—	340, 328	58JOC430
7	10, 13	OMe, NH ₂	440 (3.89), 400 (3.95), 376 (4.00), 352 (4.19)	73JOC4167
8	2, 3, 10	(OMe) ₃	435, 332	61JOC2231
9	2, 3, 10	—OCH ₂ O—, OMe	437, 342	60JA1145
10	2, 3, 9, 10	(OMe) ₄	464, 355	61JOC2231
11	2, 3, 10, 11	(OMe) ₄	417, 322	61JOC2231
12	2, 3, 9, 10	(OMe) ₂ , —OCH ₂ O—	492, 358	61JOC2231
13	2, 3, 10, 11	(OMe) ₂ , —OCH ₂ O—	413, 322	61JOC2231
14	2, 3, 9, 10	—OCH ₂ O—, (OMe) ₂	460, 348	60JA1145
15	2, 3, 10, 11	—OCH ₂ O—, (OMe) ₂	422, 326	60JA1145
16	2, 3, 9, 10	—OCH ₂ O—, —OCH ₂ O—	490, 356	61JOC2231
17	2, 3, 10, 11	—OCH ₂ O—, —OCH ₂ O—	417, 332	60JA1145
18	2, 3, 10, 9	(OMe) ₃ , OH	477, 354	61JOC2231
19	2, 3, 9, 10	—OCH ₂ O—, OH, OMe	470, 352	61JOC2231
20	2, 3, 9, 10	—OCH ₂ O—, OMe, OH	463 (3.15), 349 (4.35)	66TL3069
21	2, 3, 9, 10, 11	(OMe) ₄ , OH	438 (3.78), 415sh (3.69), 367sh (3.71)	69JOC1349
22 ^b	2, 3, 10, 11, 8	(OMe) ₄ , Me	420 (3.89), 400 (3.95), 376 (4.00), 352 (4.19)	76JMC882
23 ^c	2, 3, 10, 11, 6	(OMe) ₄ , Me	425 (4.27), 410 (4.22), 360 (3.98)	76JMC882
24 ^d	2, 3, 11, 12, 8	(OMe) ₄ , Me	451 (4.08), 430 (4.13), 410 (4.15)	76JMC882

^a Only the first and second bands, or the bands, $\lambda_{\max} > 350$ nm, are shown.

^b Neocoralyne.

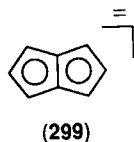
^c Isocoralyne.

^d Allocoralyne.

TABLE XII
ABSORPTION SPECTRA OF POLYCYCLIC AROMATIC NITROGEN CATIONS

Compound	λ_{\max} nm (log ϵ)	Refs.
23	464 (3.59), 426 (4.30), 404 (4.11)	60JOC191
24	462 (3.67), 433 (3.69), 410 (4.23)	60JOC191
25	415 (3.60), 395sh, 388 (4.54)	64JOC856
26	422 (3.73), 384 (4.59)	64JOC856
27	427 (3.70), 404 (3.82)	64JOC856
30	394 (4.10), 374 (4.03)	56JA2459
33	455sh, 409 (4.16), 391 (4.03)	65JOC1846
180	417 (4.07), 395 (3.96)	87JCS(P1)481
195	433 (3.76), 410 (3.69)	89CS367
199	456sh (3.70), 432 (4.36), 410 (4.18)	68JOC390
204	435 (4.26), 412 (4.06)	87JCS(P1)481
206	435 (4.03), 413 (3.87)	89TL7217
208	432 (3.90), 410 (3.82)	80JCS(P1)1879
210	434 (4.36), 406.5 (4.56)	68JHC253
211	439sh (4.03), 416.5sh (4.47), 409 (4.48)	68JHC253
212	461 (4.22), 433 (4.08), 407 (3.82)	68JHC253
221	440 (4.43), 419 (4.35)	65JOC252
227	446 (3.98), 420 (3.99)	91BCJ1996

ii. *Example 2: Pentalene dianion (299), 4-azapentalenyl anion (41), and 4,8-diazapentalene (42).* Compounds **41** and **42** are isoelectronic, with **299** having 10π electrons. The maximum absorption band at 295 nm and a shoulder at 210 nm of **41** are assigned to the bands mainly contributed by HOMO–LUMO and HOMO–next-LUMO transitions, respectively. For **42**, a band with the maximum at 284 nm has been assigned to the transition ${}^1A_g - {}^1B_{3u}$, mainly due to a combination of excitations of HOMO–next-LUMO and next-HOMO–LUMO. Using Eq. (10), the bands of **41** and **42** can be assigned to those of the parent hydrocarbon **299**. The calculated results are compared in Table XIV.



iii. *Example 3: Indolizine (43), pyrazolo[1,2-a]pyridazinium ion (44), and indole anion (47).* Compounds **43** and **44** are isoelectronic with **47**. The shifts of the transition energies of indole by replacing a bridgehead

TABLE XIII
ABSORPTION SPECTRA OF ACENAPHTHYLENE (**298**) AND ITS AZONIA DERIVATIVE (**237**)

Transition ^a (weight %)	298			237	
	Calculated ^b $\lambda(\text{nm})$ (f) ^d	Observed ^c $\lambda(\text{nm})$ log(ϵ)		Calculated ^b $\lambda(\text{nm})$ (f) ^d	Observed ^c $\lambda(\text{nm})$ log(ϵ)
6-7 (77)(+) x	384 (0.02)	376 (2.2)	→	6-7 (68) x	365 (0.07) 332 (3.79)
4-7 (55) (+ +) x	314 (0.16)	322 (4.0)	→	5-7 (69) y	345 (0.06)
5-7 (92) (±) y	309 (0.28) ^e	275 (3.5)	→	6-8 (71) y	251 (1.00) 295 (3.58)
6-8 (85) (−) y	248 (0.00)		→	5-8 (60) x	246 (1.46) 229 (4.81)
5-8 (68) (−) x	220 (1.31)	230 (4.7)	→	4-7 (81) x	208 (0.49)

^a Signs in parentheses are the expected shift of the transitions by replacement of a carbon atom with a nitrogen atom; (+), hypsochromic shift and (−), bathochromic shift. The directions of transition are denoted by x and y.

^b M. Hida, unpublished data.

^c Ref. 84JCS(P1)2553.

^d Oscillator strength.

^e Although the shift of the transition (5-7) is slight, the bathochromic shift of the band mainly contributed by the transition (5-7) is caused by the configuration interaction (CI) of the second-order perturbation with the transition (6-8).

TABLE XIV
CALCULATED ABSORPTION SPECTRA OF PENTALENE DIANION AND AZAPENTALENES

Calculated λ_{\max} (nm) (oscillator strength)		
299 ^a	41 ^b	42 ^b
268 (0.345)	264 (0.250)	289 (0.280)
234 (0.060)	252 (0.040)	265 (0.091)
215 (0.000)	211 (0.037)	216 (0.000)

^a M. Hida, unpublished results

^b Ref. 68TCA(11)411.

carbon atom with a nitrogen atom can be estimated using Eq. (10). The shifts of the absorption bands after the CI procedure can be qualitatively estimated by the differences of the transition energies (ΔE_{i-j}) of the component transitions ($i - j$) and their weights in CI as

$$\Delta E \sim \Sigma (\text{weight } \%) \Delta E_{i-j} \quad (11)$$

In Table XV, the calculated and the observed bands are compared, along with the qualitative estimation of the shifts of the transitions and the absorption bands of **47**.

Table XVI reveals that the spectra of **43** having nitrogen atoms at positions 1 and 3 hypsochromically shift from that of **43**, as expected using the developing coefficients of the HOMO and LUMO obtained by the simple HMO method. However, the spectra of derivatives having a nitrogen atom at other positions do not exhibit significant shifts from that of parent **43**.

2. NMR Spectra

a. ¹H-NMR Spectra. Sanders and co-workers examined the ¹H-NMR spectra of parent quinolizinium ion (**1**) and first completed the assignment [81H(15)213]. A characteristic down-field shift ($\sigma = 9.58$) is observed for protons at the 4- and 6-positions because of the adjacent quaternary nitrogen. The coupling constants are $J_{12} = 8.5$ Hz and $J_{23} = 7.2$ Hz [84JCS(P1)2553]. Table XVII shows the chemical shifts of substituted quinolizinium salts. In the case of the compounds with an electron-donating group at the 1-, 2-, or 4-position, the protons at 2- and 4-, 1- and 4-, or 1- and 3-positions shift upfield, respectively. The characteristic proton at the 6-position of **2** and its derivatives gives a singlet in the region $\delta = 10.6$ –11.0. Bradsher and co-workers reported that the chemical shifts

TABLE XV
 ABSORPTION SPECTRA OF ISOCONJUGATED SYSTEMS OF THE INDOLE ANION

Compound	Transition ^a nm	After CI nm	Weight (%)	f ^b	Observed $\lambda_{\max}(\log \epsilon)$
47^c	5-6 344(±)	365(+)	5-6:94 4-7: 3.5	0.061	<i>d</i>
	4-6 255(+)	288(+)	4-6:58.2 5-7:37.2	0.013	
	5-7 246(-)	230(-)	4-6:35.1 5-7:60.6	1.317	
	4-7 211(+)	217(+)	4-7:78.4 5-8:12.7	0.107	
	5-8 190(±)				
43^c		348	5-6:89	0.092	346 (3.29)
		288	5-7:59 4-6:34	0.043	295 (3.56)
		213	4-7:54 3-6:16 5-8:14	0.134	238 (4.51)
		212	4-6:50 5-7:27	1.095	
44^f		333	5-6:81 4-7:17	0.07	<i>d</i>
		302	5-7:80 4-6:15	0.21	
		219	4-7:68 5-6:14 5-8:13	0.47	
		205	4-6:66 5-7:14 3-6:11	1.09	

^a Signs in parentheses are the expected shift by replacement of a carbon atom with a nitrogen atom; (+), the hypsochromic shift; (±), the small shift.

^b Oscillator strength.

^c M. Hida, unpublished results.

^d Not observed.

^e Ref. 68TCA(9)222.

^f Ref. 69T2259.

of H-6 of 9-substituted **2** correlates with Hammett substituent constants (σ_p) (73JOC2917).

¹H-NMR spectra of all the isomers of (monomethyl)benzo[*a*]quinolizinium salts were examined [92JHC215]. The methyl groups appeared down field compared to the corresponding methyphenanthrenes, and the shifts at the 2- and 4-positions are larger than the others.

TABLE XVI
SHIFTS OF THE FIRST ABSORPTION BAND OF AZA-INDOLIZINES

	Position of nitrogen atom of Aza-indolizine ^a							
	1	2	3	5	6	7	8	9
HOMO	0.493	0.069	0.515	0.401	0.222	0.330	0.327	0.226
LUMO	0.254	0.213	0.072	0.571	0.192	0.415	0.529	0.014
Shift (theor.) ^b	(+)	(±)	(+)	(-)	(±)	(±)	(-)	(±)
Shift (obs.) ^c	0.42	0.02	0.54	<i>d</i>	<i>d</i>	0.08	<i>d</i>	<i>d</i>

^a Numbering is shown in compound **43**.

^b Signs in parentheses are the expected shift by replacement of a carbon atom with a nitrogen atom; (+), the hypsochromic shift; (-), the bathochromic shift.

^c Shift (eV) from indolizine [68TCA(9)222].

^d Not observed.

Recent advancements in 2D-NMR spectroscopy make it possible to assign all the signals of complex molecules. By the COSY method, all the protons of **243** were assigned (89H2369). The complete ¹H assignment has also been reported for 6-methyl-2,3,10,11-tetramethoxydibenzo-[*a,g*]quinolizinium chloride (76JMC882), **208** (83OMR649), and **237** (84JCS(P1)2553).

TABLE XVII
CHEMICAL SHIFTS IN ¹H-NMR SPECTRA OF SUBSTITUTED QUINOLIZINIUM SALTS
IN DMSO-d₆

Substituent	Positions								Refs.
	1	2	3	4	6	7	8	9	
H	8.69	8.43	8.14	9.59	9.58	8.14	8.43	8.69	81H(15)213
2-Br	9.07	—	8.36	9.42	9.51	8.13	8.41	8.54	81H(15)213
2-OH	7.68	—	7.62	9.23	9.06	7.63	7.99	8.23	81H(15)213
2-NC ₅ H ₁₀	7.57	—	7.62	8.83	8.63	7.20	7.64	7.82	81H(15)213
2-NMe ₂	7.44	—	7.51	8.89	8.72	7.24	7.68	7.94	81H(15)213
4-Br	8.79	8.59	8.34	—	9.69	8.25	8.52	8.74	81H(15)213
4-NC ₅ H ₁₀	8.42	8.42	7.81	—	9.23	8.12	8.36	8.66	83OMR220
4-NMe ₂	8.26	8.46	7.89	—	9.54	8.12	8.36	8.56	82JHC797
3-Me	8.49	8.16	—	9.56	9.50	7.96	8.24	8.53	86CB2062

Katritzky and co-workers reported the pseudo-base formation of **208** and its derivatives by using ^1H -NMR spectra. Spectra of DMSO- d_6 samples are sensitive to the presence of water and an up-field shift was observed. They suggested covalent hydration at position 2 (83OMR649).

b. ^{13}C -NMR Spectra. ^{13}C chemical shifts for quinolizinium salts were reported (83OMR220; 86CB2062). The effect of the quaternary nitrogen was also observed in ^{13}C NMR (Table XVIII). The carbon atoms at the 4- and 6-positions appear to be about 137 ppm, and the other carbons appear near 125 ppm. The other characteristics of ^{13}C NMR are the broadening of C-4 and C-6 due to ^{13}C - ^{14}N coupling; moreover, $^1J(\text{CH})$ values for C-4 and C-6 are larger than those for the other carbon atoms. These characteristics are useful for the assignment of ^{13}C spectra. A few ^{13}C -NMR shifts are available for polycyclic nitrogen cations **208** and its derivatives (83OMR649) and **237** [84JCS(P1)2553].

c. ^{15}N -NMR Spectra. The ^{15}N chemical shifts for **1** are reported to be 207.1 ppm (relative to anhydrous ammonia) [84JCS(P1)2553] and 171.4 ppm (relative to nitromethane) (89MI1). Other spectral data are also obtained (91JOC4858): **39** (199.6 ppm), **237** (184.4 ppm), and **246** (195.2 ppm).

3. Mass Spectra

Mass spectra of aromatic nitrogen cations using the electron impact ionization technique do not give any useful information because of the low volatility and thermal degradation of the nitrogen cations. Recent developments in mass spectroscopy allow the measurement of quaternary nitrogen cations [84JOC764; 87JCS(P2)633]. Fast atom bombardment (FAB) and field desorption (FD) techniques are quite useful for measuring molecular ions of polycyclic aromatic nitrogen cations. For example, the photocyclization of **202** gave not the 2*a*-azoniadibenzo[*c,g*]phenanthrene salt, but 2*a*-azoniabenzo[*g,h,i*]perylene salt (**204**) by double-cyclization. This result was supported by mass spectra using the FD technique that showed a peak at m/z 278 corresponding to the naked cation of **204** [87JCS(P1)481]. On FAB, the MS spectrum of 2*a*-azoniabenzo[*a*]coronene salt (**227**) using *m*-nitrobenzylalcohol as a matrix contained the cation at m/z 352 as the highest m/z value (91BCJ1996).

TABLE XVIII
¹³C-NMR CHEMICAL SHIFTS OF SUBSTITUTED QUINOLIZINIUM SALTS IN DMSO-d₆

Substituents	1	2	3	4	6	7	8	9	9a	Refs.
H	127.9	138.0	125.0	137.0	137.0	125.0	138.0	127.9	143.0	83OMR220
1-Br	121.8	141.2	124.5	137.0	138.5	125.7	140.0	127.3	142.3	83OMR220
2-Br	130.1	133.2	128.4	137.4	137.4	125.2	139.2	127.1	143.4	83OMR220
2-OH	108.9	164.4	117.0	139.2	135.5	120.8	135.9	125.7	145.9	83OMR220
2-NC ₅ H ₁₀	102.2	152.3	111.5	136.8	133.8	117.6	134.0	124.9	144.1	83OMR220
2-NMe ₂	100.8	150.9	110.7	136.2	133.2	116.8	133.2	124.2	143.3	83OMR220
3-Me ^a	126.1	139.0	134.6	134.0	135.1	123.6	135.7	126.6	140.5	86CB2062

^a Solvent: CDCl₃ + 20% DMSO-d₆.

C. ELECTROCHEMICAL PROPERTIES

Aromatic hydrocarbons are called π bases because of their rather low ionization potentials. On the other hand, aromatic compounds containing a nitrogen atom possess lower MO energy than the parent hydrocarbons, and they show less electron-donating character or electron-accepting character. The mode of action of the herbicide paraquat **300** (1,1'-dimethyl-4,4'-bipyridinium salt) is thought to be connected with its reversible reduction-oxidation reactivity. The compounds with a lower level of activity possess higher reduction potentials, and their one-electron transfer is not quite quantitatively reversed.

In the following sections, the reduction potential and the electrochemical stability of the one-electron reduced species (semiquinone intermediate) are discussed using MO theory.

1. Reduction Potentials

Streitwieser showed that the LUMO energy calculated by the simple HMO method is linearly well correlated to the reduction potential (61MI2). Since the electron affinity of π -electron systems equals the LUMO energy obtained by the SCF MO method (33MI1), the reduction potential can be thought to be proportional to the LUMO energy. Solvation energy may largely influence the reduction potential because of the large change in charge on electron transfer. Combining the LUMO energy (ϵ_{lumo}) with the solvation energy (W_s), the one electron reduction potential (E_1) can be written as

$$E_1 = a\epsilon_{\text{lumo}} + b + W_s \quad (12)$$

where a and b are constants. Using the Born equation (20MI1), the solvation energy can be written in terms of Eqs. (13)–(15), where c is a constant, and r and D are the Onsager's radius and the dielectric constant of the solvent, respectively.

Aromatic hydrocarbons: $A + e \rightarrow A^-$

$$W_s^0 = c \frac{e^2}{r^2} \left(\frac{1}{D} - 1 \right) \quad (13)$$

Aromatic nitrogen monocation: $A^+ + e \rightarrow A$

$$W_s^0 = -c \frac{e^2}{r^2} \left(\frac{1}{D} - 1 \right) \quad (14)$$

Aromatic nitrogen dication: $A^{++} + e \rightarrow A^+$

$$W_s^0 = -3c \frac{e^2}{r^2} \left(\frac{1}{D} - 1 \right) \quad (15)$$

The linear correlation of reduction potentials of 13 aromatic hydrocarbons in acetonitrile with the values of ϵ_{lumo} can be written by Eq. (16) (9IUP1).

$$E_1(\text{CH}_3\text{CN, vs. SCE}) = -0.768\epsilon_{\text{lumo}} - 3.983. \quad (16)$$

The reduction potentials of the aromatic nitrogen cations are expected to deviate from Eq. (16), where the deviation of the monocation is $2W_s^0$ and that of the dication is $4W_s^0$. The results compared in Table XIX are very reasonable, and the Onsager's radius of the solvent is calculated to be about 6 Å from the value of W_s^0 .

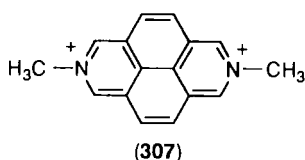
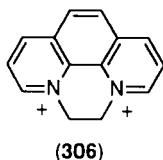
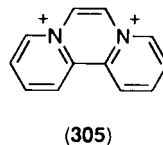
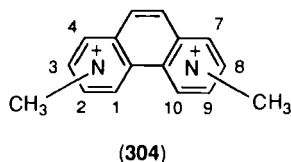
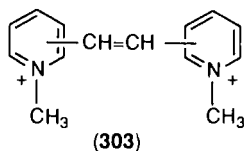
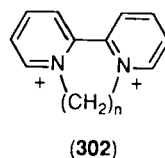
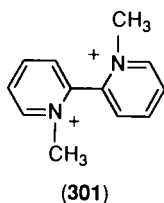
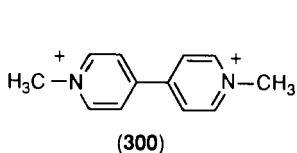


TABLE XIX
REDUCTION POTENTIALS OF AROMATIC NITROGEN CATIONS^a

Compound	ϵ lumo (eV)	E_{calc} (V)	E_{obs} in CH ₃ CN (vs.SCE)	Difference ^b (V)
Phenanthrene	-2.0713	-2.39	-2.75	0.36
3	-6.6604	1.13	-1.18	2.31
4	-6.7405	1.19	-1.06	2.25
16	-6.4780	0.99	-1.18	2.17
17	-6.6015	1.09	-1.02	2.11
204 (R = H)	-6.5825	1.03	-1.07	2.10
300	-10.9542	4.43	-0.40	4.83

^a Arai and Hida, unpublished results.

^b Mean of the differences between calculated and observed values of monocations is 2.19 V.

Roughly speaking in terms of first-order perturbation theory, the differences in the LUMO energy between the aromatic nitrogen cations and their parent hydrocarbons are proportional to the number of nitrogen atoms. The solvation energy also influences the reduction potentials in proportion to the number of nitrogen atoms. As shown in table XIX and XX, the reduction potentials are roughly determined by the number of nitrogen cations; those of the aromatic hydrocarbons are -2 to -3 volts, and those of the monocations and the dications are -1 to -2 and -0.3 to -0.5 volts, respectively.

2. Disproportionation Reactivity of One-Electron Reduced Species

The one-electron reduced species (semiquinone intermediates) undergo disproportionation reaction [Eq. (17)].



The equilibrium constant (K) can be determined by the difference between the potentials (E_1 and E_2) of the first and second steps of the reduction only when they are reversible (35CR243).

$$K = \frac{[A^{\cdot -}]^2}{[A][A^-]} \quad (18)$$

$$E_1 - E_2 = 0.059 \log K \quad (19)$$

In direct current polarography, it has been shown that both of the two-reduction waves ($A \rightarrow A^{\cdot -}$ and $A^{\cdot -} \rightarrow A^-$) can be observed when K is

TABLE XX
 REDUCTION POTENTIALS OF AROMATIC NITROGEN DICATIONS

Compound	Reduction	Potential ^a (volt)	ϵ_{lumo}^b
25	-0.40	-0.78	- 9.91
26	-0.40	-0.78	- 9.73
27	-0.31	-0.58	- 9.83
159	-0.45 ^c	-0.88 ^c	-10.29
300	-0.40	-0.82	-10.95
301	-0.68	-0.75	-10.70
302 ($n = 2$)	-0.34	-0.82	-10.63
302 ($n = 3$)	-0.52	-0.85	-10.71
303 (4,4')	-0.45	-0.66	- 9.93
303 (2,2')	-0.47	-0.66	-10.70
303 (2,4')	-0.46	-0.66	-10.00
304 (3,8)	-0.37	-0.84	-10.44
304 (2,9)	-0.45	-0.72	-10.45
305	-0.21	-0.88	-10.49
306	-0.25	-0.64	-10.54
307	-0.44	-0.93	-10.22

^a Versus Ag/AgCl, Solvent: MeCN (73LA324, 73LA339).

^b M. Hida, unpublished results.

^c Solvent: DMF.

larger than 10^3 (55JCP514). Molecular orbital theory shows that the energy difference ($E_1 - E_2$) can be connected with the electron repulsion energy (J) between two electrons occupying a LUMO (55JCP514).

$$J = \sum_{r,s} C_{r,\text{lumo}}^2 C_{s,\text{lumo}}^2 (rr | ss) \quad (20)$$

In the case of aromatic hydrocarbons, the value of $\log K$ is apparently proportional to the value of J calculated using the PPP method (91UP1).

$$\log K = 9.2J - 32.6 \pm 5.2 \quad (21)$$

The semiquinone may be electrochemically stable when J is larger than 4 eV. As compared in Table XXI, the calculated values of K using Eq. (21) agree with the observed values with several exceptions. The J value of diquat (**302**; $n = 2$) is calculated by assuming that the dihedral angle between the two pyridinium rings is 10 degrees, which has been concluded from the CNDO/2 MO method (86CJC1711). The calculated K values for **305** and **307** are unexpectedly lower than those observed, while the calculated K of **301** is very much larger than the observed value.

TABLE XXI
OBSERVED AND CALCULATED VALUES OF FORMATION CONSTANTS (K) AND VALUES OF
ELECTRON REPULSION ENERGY (J)

Compounds	log <i>K</i>		Calculated ^b	<i>J</i> (eV) ^b
	Observed ^a			
	DMF	CH ₃ CN		
300	7	7	7.0	4.30
301	0.1	1.3	7.1	4.31
302 (<i>n</i> = 2)	7.6	8	7.1	4.31
302 (<i>n</i> = 3)	5.5	5.9	7.0	4.30
303 (4,4')	3.3	3.6	3.3	3.9
303 (2,2')	2.9	3.3	3.7	3.95
303 (2,4')	3.6	3.3	3.6	3.93
304 (3,8)	7.8	8.0	6.0	4.19
304 (2,9)	5.6	4.6	5.1	4.10
305	9.8	11.3	6.1	4.21
307	8		4.4	4.02

^a Refs. 73LA324, 73LA339.

^b M. Hida, unpublished results.

3. Electrochromic Character

Most of the reduced polycyclic compounds are colored. The polycyclic aromatic nitrogen cations have lower reduction potentials, and their reduced species can absorb light in the visible region. A representative compound having the electrochromic character is viologen. As shown in Table XXII, the reduced species of **300** absorbs light at about 600 nm with a strong intensity and is colored deep blue. The reduced species of **17** is colored green, but its absorption is rather weak.

TABLE XXII
ABSORPTION SPECTRA OF REDUCED SPECIES^a

Compound	λ_{max} (nm)	
	Observed	Calculated (oscillator strength)
17	613	656 (0.04), 512 (0.47)
300	608, 402	657 (1.23), 455 (0.02)

^a Arai and Hida, unpublished results.

VI. Reactivity

Many examples of the reaction of quinolizinium and benzoquinolizinium salts are described in two reviews by Jones [82AHC(31)1] and Bradsher (84MI1). Hence in this chapter the nucleophilic and electrophilic reactions are discussed in terms of MO theory. The reactivities of substituents are also described.

A. SUBSTITUTION REACTION

Orientation in electrophilic and nucleophilic reactions of aromatic compounds can be predicted with the aid of the reactivity index of MO theory. Electrophiles will attack positions of higher electron densities, larger superdelocalizability (electrophile), and the lower localization energy (electrophile). On the other hand, nucleophilic attack is preferred at positions of lower electron densities, larger superdelocalizability (nucleophile), and lower localization energy (nucleophile). Table XXIII shows reactivity indexes of some aromatic nitrogen cations.

1. *Electrophilic Reaction*

Pyridinium, quinolinium, and isoquinolinium cations are the major species undergoing electrophilic substitution reactions under acidic conditions [90AHC(47)1]. As expected from Table XXIII, the electrophilic reaction of pyridinium ion occurs at the 3-position, and an electrophile attacks at the 5- and 8-positions of quinolinium and isoquinolinium cations. Electrophilic reactivity of **1** is rather low because of its electron accepting character. Molecular orbital calculations of its orientation did not give a consistent conclusion. Electron density and superdelocalizability (electrophile) predict that position 1 will be the most reactive towards an electrophile, while inspection of the localization energy (electrophile) predicts that electrophilic reaction takes place at position 4.

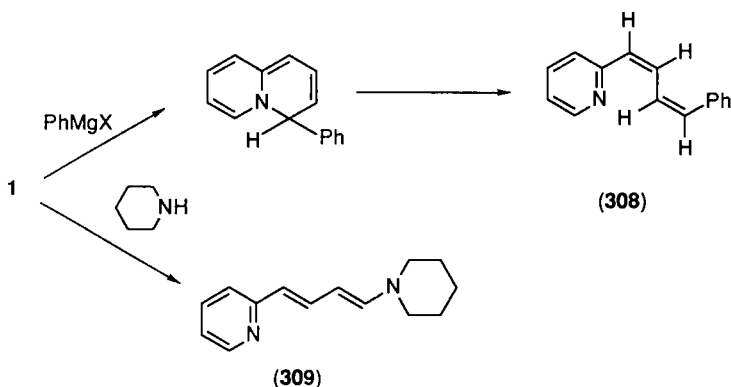
Only a few examples are available regarding electrophilic substitution of parent **1** and benzoquinolizinium salts (**2**, **3**, and **4**). The reaction of **1** with bromine yielded the perbromide, which gave 1-bromoquinolizinium bromide in 69% by heating at 200°C [81H(15)213]. The electrophilic reaction of benzoquinolizinium salts takes place at the ring without the quaternary nitrogen. This result is similar to the fact that electrophiles attack the 5- and 8-positions of quinoline and isoquinoline. Compound **4** was nitrated (36%) with $\text{HNO}_3\text{--H}_2\text{SO}_4$ to yield a 10-nitro derivative and was sulfonated with fuming sulfuric acid at position 10 to give betaine (69%)

[71JCS(C)3650]. The reaction of **3** with $\text{HNO}_3\text{--H}_2\text{SO}_4$ at -5°C yielded a 10-nitro derivative (65.5%) (74JOC1157). Sulfonation occurred at position 10 to give the betaine (82%) (66JOC565).

2. Nucleophilic Reaction

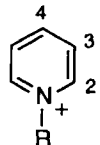
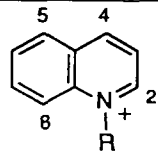
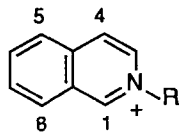
Nucleophiles, such as hydroxide, cyanide, and Grignard reagents attack the 2-position of pyridinium and the 1-position of isoquinolinium cations. Quinolinium salt reacts with nucleophiles at the 2- and 4-positions. Hydroxide attacks the 2-position, and cyanide attacks the 4-position. These results support the theoretical expectation.

All reactivity indexes predict that nucleophilic reaction takes place at position 4 of **1**. Miyadera and co-workers examined the reaction of **1** with nucleophiles, such as Grignard reagents (64CPB1344), LiAlH_4 (69T397), and NaBH_4 (69T397). These reagents attack at the 4-position of the quinolizinium ring to form intermediate 4*H*-quinolizines, which undergo ring opening to yield 4-(2-pyridyl)-1,3-butadienes **308**. The ring-opened products, pyridylbutadienes **309**, were also obtained by a reaction with piperidine, morpholine (71LA65), or diethylamine (82JHC797).



Sanders and co-workers reported the reaction of four (monobromo) quinolizinium salts with amines (82JHC797; 83JHC407). A soft nucleophile, aniline, reacts with the 2- and 4-bromo derivatives to give substitution products, a 2-anilino derivative and 4-phenylimino-4*H*-quinolizine **310**, respectively. The reaction with a harder nucleophile, ammonia, yielded the ring-opened products, for example, **311**. In the reaction of 2- and 4-bromoquinolizinium salts with diethylamine, intermediate in hardness, both reactions occur. For example, the 4-bromo derivative undergoes substitution and then slow ring-opening to give **312**. In the cases of 1- and

TABLE XXIII
REACTIVITY INDEXES^a OF AROMATIC NITROGEN CATIONS

	Position	2	3	4					
	q ^b	0.759	1.012	0.835					
	L(E) ^c	2.71	2.56	3.07					
	L(N) ^d	1.86	2.56	2.01					
	S(E) ^e	0.596	0.823	0.546					
	S(N) ^f	1.596	0.823	1.546					
	Position	2	3	4	5	6	7	8	
	q	0.694	1.022	0.788	0.961	1.008	0.950	1.033	
	L(E)	2.84	2.53	2.92	2.43	2.50	2.63	2.36	
	L(N)	1.70	2.53	1.79	2.13	2.50	2.24	2.36	
	S(E)	0.500	0.847	0.535	0.858	0.861	0.754	0.951	
	S(N)	2.278	0.847	2.313	1.302	0.861	1.198	0.951	
	Position	1	3	4	5	6	7	8	
	q	0.671	0.842	1.024	1.009	0.946	1.000	0.946	
	L(E)	2.53	2.37	2.34	2.31	2.66	2.48	2.42	
	L(N)	1.60	2.02	2.34	2.31	2.24	2.48	2.11	
	S(E)	0.536	0.819	0.970	0.984	0.736	0.873	0.855	
	S(N)	2.314	1.263	0.970	0.984	1.180	0.873	1.300	
	Position	1	2	3	4				
1 ^g	q	1.005	0.916	1.011	0.856				
	L(E)	2.31	2.62	2.50	2.27				
	L(N)	2.31	2.20	2.50	1.97				
	S(E)	0.992	0.730	0.866	0.882				
	S(N)	0.992	1.175	0.866	1.327				

	Position	1	2	3	4	6	7	8	9	10	11
2^g	q	1.004	0.939	1.013	0.877	0.791	0.968	1.000	0.968	1.005	1.013
	L(E)	2.24	2.44	2.44	2.13	2.00	2.28	2.42	2.50	2.24	2.03
	L(N)	2.24	2.25	2.44	1.99	1.67	2.14	2.42	2.30	2.24	2.03
	S(E)	1.072	0.856	0.912	1.040	1.001	0.983	0.922	0.832	1.069	1.303
	S(N)	1.072	1.106	0.912	1.290	2.001	1.233	0.922	1.082	1.069	1.303
	Position	1	2	3	4	6	7	8	9	10	11
3^g	q	1.006	0.895	1.011	0.833	0.901	1.019	1.006	0.977	1.000	0.979
	L(E)	2.37	2.68	2.52	2.36	2.21	2.32	2.32	2.56	2.45	2.42
	L(N)	2.37	2.13	2.52	1.92	2.09	2.32	2.32	2.39	2.45	2.28
	S(E)	0.936	0.686	0.853	0.803	0.987	0.983	0.972	0.806	0.893	0.883
	S(N)	0.936	1.326	0.853	1.443	1.147	0.983	0.972	0.966	0.893	1.043
	Position	1	2	3	4	5	6	7	8	9	10
4^g	q	0.826	1.014	0.895	1.005	1.010	0.906	0.984	1.005	0.978	1.021
	L(E)	2.36	2.48	2.72	2.33	2.31	2.45	2.37	2.51	2.50	2.40
	L(N)	1.92	2.48	2.14	2.33	2.31	2.03	2.24	2.51	2.35	2.40
	S(E)	0.792	0.881	0.671	0.974	0.989	0.796	0.919	0.855	0.842	0.921
	S(N)	1.432	0.881	1.311	0.974	0.989	1.436	1.079	0.855	1.002	0.921

^a All reactivity indexes are calculated by HMO.

^b Electron density.

^c Localization energy for electrophile.

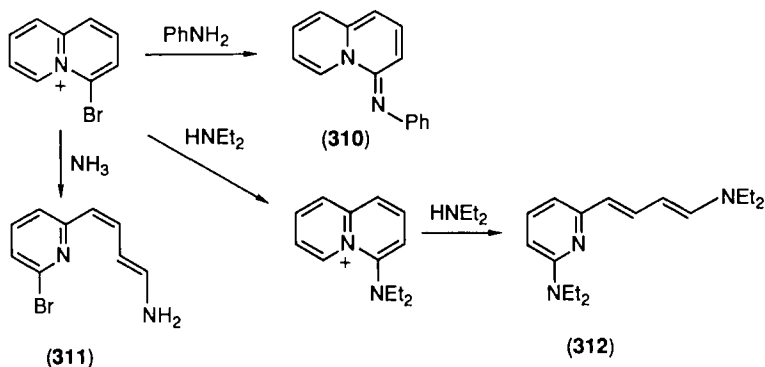
^d Localization energy for nucleophile.

^e Superdelocalizability for electrophile.

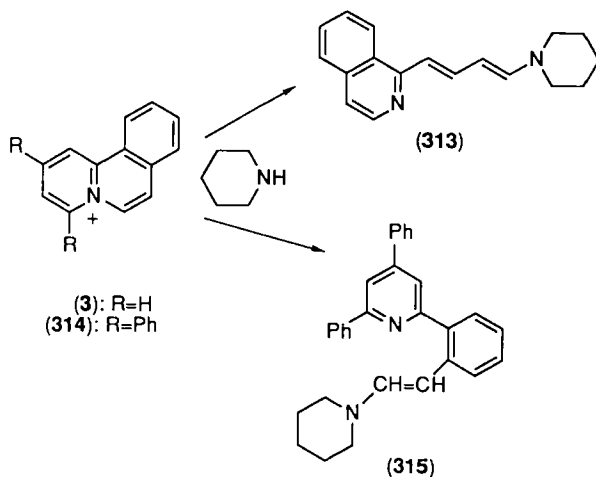
^f Superdelocalizability for nucleophile.

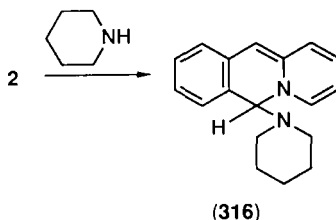
^g Numberings are shown in text.

3-bromo derivatives, ring-opened products were obtained. Alaimo *et al.* obtained many derivatives of 2-arylaminoquinolizinium salts by a reaction between 2-bromo quinolizinium salt and arylamines and examined their activities as anthelmintic agents (70JMC554).



The reaction of **3** and **4** with piperidine gave the ring-opened products isoquinolybutadiene **313** and quinolybutadiene in 76% and quantitative yields, respectively (71LA65). With 2,4-diphenylbenzo[*a*]quinolizinium salt (**314**), piperidine attacks position 6 to give 2,4,6-triphenylpyridine derivative **315** (76%) [81H(15)1175]. In the reaction of **2** with piperidine at the 6-position, 6*H*-benzoquinolizine **316** is formed (71LA65).





Localization energy (nucleophile) suggests that benzoquinolizinium cations (**2**, **3**, and **4**) are more reactive than **1**. Salt **1** does not react with hydroxide and cyanide ions, while hydroxide ion attacks the 6-position of **2** and the 1-position of **4** [58JCS3067; 67JOC733; 71JCS(C)3650]. The reaction of **2** with cyanide ion is also reported (58JCS3067). In the reaction of colaryne (**110**), hydroxide attacks the 8-position to yield ring-opened product (75JPS1825).

B. ACIDITIES OF HYDROXY GROUP COMBINED WITH THE QUINOLIZINIUM RING

Acidities of hydroxyquinolizinium cations can be predicted using both a resonance effect (E_E) and a solvation effect (E_S).

$$(\text{p}K_a)_{\text{calc}} = E_E + E_S \quad (22)$$

The resonance effect on acidity can be expressed in terms of the electron density (q_0) at the oxygen atom as

$$E_E = 44.5q_0 - 76.64 \quad (23)$$

where the coefficients are determined from the linear relationship between the acidities of hydroxy-substituted aromatic hydrocarbons and their electron densities obtained by the HMO-omega method. The solvation effect can be determined using a Westheimer model (38JCP506) in which solutes are assumed to be included in spheres having a different dielectric constant from that of outer solvent. As shown in Table XXIV, the calculated $\text{p}K_a$ values are in agreement with the experimental results, and largely depend on solvation effects rather than on resonance effects (78NKK1249).

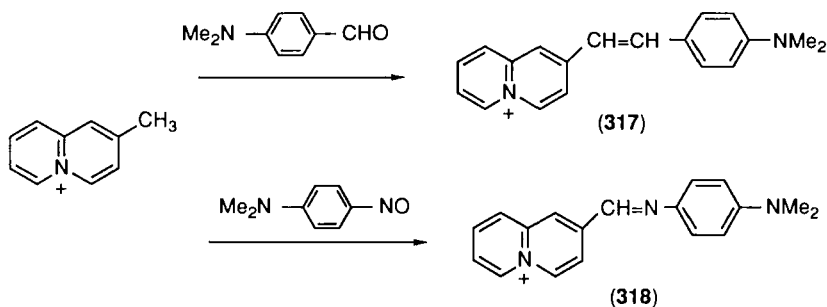
TABLE XXIV
OBSERVED AND CALCULATED VALUES OF pK_a OF HYDROXYQUINOLIZINIUM BROMIDES^a

Position of OH	1	2	3	4
π -Electron density	1.937	1.932	1.942	1.924
Resonance effect	9.57	9.33	9.75	8.97
Solvation effect	-4.63	-3.57	-4.41	-5.54
(pK_a)calc.	4.94	5.76	5.34	3.43
(pK_a)obs.	5.03 ± 0.69	4.14 ± 0.66	5.06 ± 0.47	<2

^a Ref. 78NKK 1249.

C. REACTIVITY OF METHYLQUINOLIZINIUM SALTS

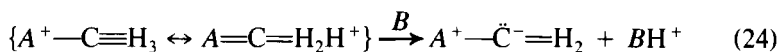
Richards and Stevens reported the reaction of 2-methylquinolizinium salt with *p*-dimethylaminobenzaldehyde and *p*-dimethylaminonitrosobenzene in the presence of piperidine to yield 2-styryl derivative **317** and anil **318**, respectively (58JCS3067). A 2-phenyl-4-methyl derivative also condensed with *p*-dimethylaminobenzaldehyde to give a 4-styryl derivative (58%), while a 4,6-dimethyl-2-phenyl derivative did not because of steric hindrance. The reaction with 2,4,6-trimethylquinolizinium salt occurred only at the 2-position (63%) (63JOC393).



If the reactivity of a methyl group in aldol or Knoevenagel condensations can be assumed to depend on the ease of release of its proton to form the carbanion, reactivity then will be predicted by inspection of acidity, theoretically estimated by the following two approaches.

1. Possibility of Hyperconjugation

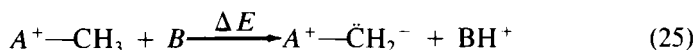
Hyperconjugation of a methyl group combined with an aromatic nitrogen cation (A^+) can be written.



When the resonance stabilization energy on the left side of Eq. (24) is large, a proton will be easily released from the methyl group by attack of base *B*. The resonance energy can be estimated by the delocalization energy, the difference between the π -electron energy of $A^+ - C \equiv H_3$ and $A^+ + C \equiv H_3$. Perturbation theory suggests that the delocalization energy is highest when a methyl group is combined with a carbon atom at the position of largest absolute value of the developing coefficient of the LUMO.

2. π Energy of Carbanion Formation

Needless to say, when the change in π -electron energy of carbanion formation (ΔE) is smaller, the methyl group can more easily release its proton on attack of base.



The value of ΔE is calculated using the HMO method by assuming the inductive model of a methyl group (61MI2).

The absolute values of the developing coefficients in the LUMO ($C_{r,lumo}$) of **3** and **4** and the changes in π -electron energies of carbanion formation ($\Delta E/|\beta|$) of their methyl derivatives are compared in Table XXV. On the basis of both values, methyl groups at the starred positions in **319** and **320** are predicted to be reactive. Arai *et al.* synthesized all isomers of monomethylbenzo[*a*]quinolizinium salts and six isomers of monomethylbenzo[*c*]quinolizinium salts and examined their reactivities with *p*-methoxybenzaldehyde [90JHC1073; 92JHC215]. They demonstrated that the experimental results are in good agreement with these theoretical expectations: 2- and 4-methylbenzo[*a*]- and 1-, 3-, and 6-methylbenzo[*c*]quinolizinium salts gave the corresponding styryl derivatives.

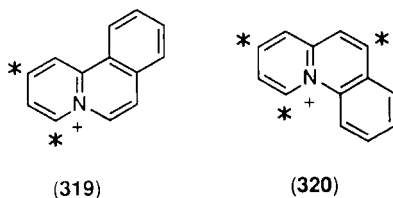


TABLE XXV
ABSOLUTE VALUES [$|C_{r,\text{lumo}}|$] OF DEVELOPING COEFFICIENTS OF THE LUMO OF BENZOQUINOLIZINIUMS AND THE CHANGES IN π -ELECTRON ENERGIES
[$\Delta E/\beta$] OF CARBANION FORMATION OF THEIR METHYL DERIVATIVES

Benzo[a]quinolizinium:										
Position (r)	1	2	3	4	6	7	8	9	10	11
$ C_{r,\text{lumo}} $	0.122	0.455	0.099	0.408	0.069	0.329	0.268	0.220	0.162	0.298
$\Delta E/ \beta $	1.2307	1.3272	1.1876	1.3527	1.2829	1.2603	1.2492	1.2280	1.2043	1.2664
Benzo[c]quinolizinium:										
Position (r)	1	2	3	4	6	7	8	9	10	11
$ C_{r,\text{lumo}} $	0.261	0.203	0.351	0.048	0.202	0.462	0.318	0.141	0.255	0.254
$\Delta E/ \beta $	1.3480	1.2047	1.3220	1.2498	1.2593	1.3686	1.2842	1.1862	1.2444	1.2303

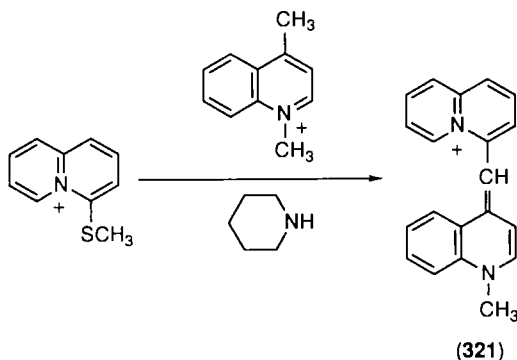
VII. Useful Applications of Polycyclic Aromatic Nitrogen Cations

A. BIOLOGICAL PROPERTIES

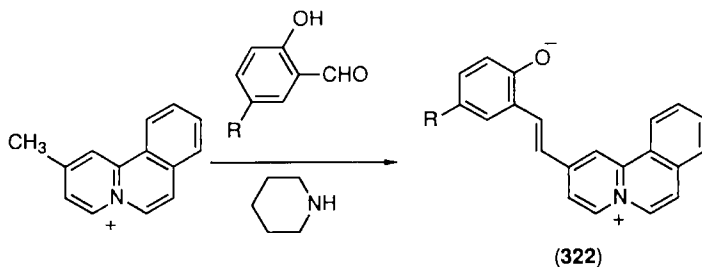
Alkaloids incorporating a quinolizinium ring are known: sempervirine, flavocorynanthrine, coralyne, and so on. Many alkaloids, such as palmatine, cryptopleurine, corydaline, and columbamine, have the dihydro form of the quinolizinium ring. From these results, many reports on the synthesis of polycyclic aromatic nitrogen cations have been published, and their biological properties have been examined (81JHC223; 84MI1).

B. DYES

The reaction of 4-methylthioquinolizinium salt with 1,4-dimethylquinolizinium salt in the presence of piperidine gave a reddish-purple cyanine dye **321** (41%) (54JOC499). Cyanine-type dyes were also obtained by the



reaction of 2-methyl quinolizinium salt or 2-methylbenzo[*a*]quinolizinium salt with *p*-dimethylaminobenzaldehyde derivatives (58JCS3067; 63JOC393; 83CC1037). Arai *et al.* reported the synthesis of stilbazolium cyanine-type dyes **322** by the reaction of 2-methylbenzo[*a*]quinolizinium



salts with hydroxy-substituted benzaldehydes in the presence of piperidine, and the pronounced negative solvatochromism of these dyes was described [90JCS(P1)1915]. The solvatochromism of binary solvent has been discussed (86MI1). Del Castillo and co-workers examined the effects of solvent on the fluorescence properties of quinolinizinium derivatives and polycyclic aromatic nitrogen cations (**30**, **201**, **252**, and **253**) and used these compounds as fluorescence probes for amines and DNA (85MI1; 88MI1). Compound **208** formed metal-association complexes with gold and mercury (83MI1; 84MI2).

C. OTHERS

The preparation and properties of the charge transfer salt between azonia derivative (**2** and **246**) and tetracyanoquinodimethane (TCNQ) were reported (88MI2). Wang and Jones reported that acridizinium salt underwent single crystal \rightarrow single crystal photodimerization (87T1273).

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